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Continuous Fiber Ceramic Composite Program

Phase I Final Report

Submitted to:

**U.S. Department of Energy
Argonne, IL 60439**

Submitted by:

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DuPont Lanxide Composites DMO CFCC Program

Phase I Final Report

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1.0 EXECUTIVE SUMMARY

In July of 1990, the U. S. Department of Energy embarked on a new and challenging research program aimed at developing continuous fiber ceramic composites (CFCCs) for wide spread commercialization in industrial applications. It was the DOE's intention to provide funding to the advanced composites industry to support the research and developments efforts already underway to commercialize CFCC materials. The strategy was to "team" composite manufacturers with original equipment manufacturers such that the research and development activities addressed the performance needs of the intended end-use applications and stayed focused on a path to commercialization. The DOE-CFCC Program is a multi-year, multi-phase, multi-million-dollar effort, whereby the composite manufacturers are the prime contractors and funded by phase, and the services of the original equipment manufacturers are subcontracted by the appropriate prime(s).

DuPont Lanxide Composites Inc. is a joint venture company between E. I. du Pont de Nemours & Co. and Lanxide Corporation chartered to manufacture and commercialize advanced ceramic matrix composites via their proprietary technologies. In 1991, DuPont Lanxide Composites assembled a complete team to address the requirements of Phase I of the DOE-CFCC Program. The team was led by DuPont Lanxide Composites and fully supported by the resources of Lanxide Corporation, DuPont, and four original equipment manufacturers; Foster Wheeler Development Corporation, Solar Turbines Incorporated, General Electric Power Generation, and Westinghouse Electric Corporation. DuPont Lanxide Composites and their subcontractors began their Phase I effort in June, 1992 and completed it in December, 1993.

The Phase I, Task 1.0 Applications Assessments were conducted by the four original equipment manufacturers. Foster Wheeler Development Corporation evaluated opportunities for CFCC materials in steam reforming and coal fired combined cycle power generating systems. General Electric and Solar Turbines evaluated opportunities for CFCC materials in turbine engine applications. Westinghouse evaluated opportunities for CFCC materials for hot gas particulate filtering applications. The results of their Task 1.0 Applications Assessments are contained in the following sections.

DuPont Lanxide Composites Inc. DIMOX™ Directed Oxidation Process

The Phase I, Task 3.1 Process Feasibility Demonstrations were conducted by the composite manufacturing team of DuPont Lanxide Composites and Lanxide. The team utilized the DIMOX™ directed metal oxidation process to fabricate 5 different types of CFCC materials during Phase I. Continuous Nicalon™ fiber reinforced alumina matrix composites were fabricated using an improved chemical vapor infiltration process for depositing silicon carbide interface coatings. A hybrid Nicalon™/Alumina composite with silicon carbide particulate additions was evaluated for reduced matrix microcracking, improved mechanical integrity, improved oxidation resistance, and enhanced thermal conductivity. A continuous HPZ™ fiber reinforced alumina matrix composite was evaluated for improved high temperature strength. A continuous Almax™ reinforced alumina matrix composite was evaluated for reduced matrix microcracking and improved chemical stability and oxidation resistance. Finally, a chopped Nicalon™ fiber reinforced alumina matrix composite was evaluated for a potential low cost fiber preforming approach. The results from the Task 3.1 Process Feasibility Demonstrations are presented here.

- A new, safe, scaleable CVI process was utilized to deposit protective SiC interface coatings in the commercially available NicalonTM/Alumina composite. These composites exhibited improved interface coating uniformity and mechanical properties equal to or better than the target values. This NicalonTM/Alumina composite provides the strength, toughness, and environmental stability required for the intended end-use applications and will be scaled-up during Phase II of the DOE-CFCC Program.
- Process feasibility was demonstrated on a hybrid NicalonTM/Alumina composite that contained ~10 vol.% SiC particulate. The incorporation of SiC particulate into the NicalonTM/Alumina composite resulted in a 25% increase in the thru thickness thermal conductivity. In addition, microstructural evaluation indicated that the SiC particulate additions significantly reduced the amount of microcracking in the matrix-rich regions of the composite. However, the observed reduction in matrix microcracking did not appear to increase the matrix microcrack yield strength.
- Process feasibility was demonstrated on an HPZTM/Alumina composite that contained ~35 vol.% HPZTM fibers in a 2-D, 0/90°, 8 HSW architecture. However, this composite exhibited low flexural strengths due to the strong bond between the BN interface coating and the HPZTM fibers. This strong interface is attributed to reactions between the oxygen rich fiber surface and the BN coating. Attempts to minimize or eliminate this interaction have not been successful to date.
- Process feasibility was demonstrated on an AlmaxTM/Alumina composite that contained ~35 vol.% AlmaxTM fibers in a 2-D, 0/90°, 8 HSW architecture. This

composite exhibited good room temperature strength and toughness, but reduced properties with increasing test temperature. The low composite strength and toughness at elevated temperatures, which suggests the presence of low melting point grain boundary phases.

- Process feasibility was demonstrated on a chopped NicalonTM/Alumina composite that contained 10 to 15 vol.% of the chopped fiber. As expected, the low fiber volume fraction resulted in low composite strengths. However, the room temperature flexural strengths were retained at 1200°C in air.

Westinghouse Hot Gas Filtration Systems

Hot gas cleaning systems provide a low risk opportunity to enhance development of a U.S. continuous fiber reinforced ceramic composite (CFCC) manufacturing base. Development of a porous CFCC filter element will beneficially support national programs, by improving control of industrial pollutants and by improving the economics of generation of power using advanced fossil fuel power plants.

The benefit of a CFCC hot gas filter for the industrial sector is its potential robustness and chemical inertness over a wide range of conditions that are representative of industrial process gas streams. A strategic feature of the Westinghouse/DLC commercialization plan is to leverage Westinghouse's current participation in the development of hot gas filter (HGF) systems for advanced fossil based utility power generation, to provide CFCC filter testing and qualification. This will accelerate acceptance by the industrial sector and reduce risk and development cost.

A highly efficient, robust, long-lived filter system capable of operating at temperatures up to 1800°F is an essential element of highly efficient combined cycle plants burning coal. The filter system is necessary not only to reduce pollutants, but also to lower the risk of eroding and corroding vital turbine parts. The system must operate at as high a temperature as practical otherwise plant efficiency is seriously impacted. Westinghouse is a key player in this critical technology by providing HGF systems and turbines, as well as combustors.

Materials Evaluation

Samples of 1/8" thick laminates made by the DuPont Lanxide Dimox™ directed metal oxidation process consisting of 2D Nicalon™ fiber/Alumina matrix were subjected to porosity tests and were found to have no detectable flow of gas through them even when subjected to a DP of 14.5 psi. Since this material could not be made porous enough to serve as a filter, it was dropped from further evaluation and attention was devoted to the DuPont Lanxide CVI SiC/SiC CFCC candidate material.

Samples of a prototype element were subjected to C-ring compression and tension tests and the results, which were found to be substantially higher than for current monolithic filters (1,500-3,500 psi).

The prototype CVI SiC/SiC candle filter element from DuPont Lanxide Composites was also subjected to testing as a filter element in Westinghouse's high temperature/high pressure loop. The initial prototype performed well as a filter with <1 ppm of dust in the outlet — well within the nominal specifications for Westinghouse HGF requirements. The pressure drops and flows were also well within their specification for filter elements.

Because the initial results are very encouraging, further development of this filter will be conducted in Phase II of this program.

Filter Element

The analysis indicates that the DuPont CVI SiC/SiC material at least has the right mix of thermal and mechanical properties to be a good starting point for a durable candle filter element. Additionally, we can observe that the CFCC material has twice the fracture toughness of the current monolithic filter which would indicate a higher level of durability.

A commercial scale 250 MWe integrated gasifier combined cycle (IGCC) plant would have a gas flow of 28,000 acfm and would require about 5,740 sq. ft. of filter surface or about 1980 candles of current size and geometry. If the CFCC candle costs 50% premium (i.e. \$225) over today's monolithic candle then the additional material cost would be \$450K. However, the replacement requirements would be reduced. If we assume that the current ceramic candle charge needs to be replaced when 15% of the candles break, then we can estimate the cost of each replacement event for current ceramic candles.

Cost of candle @ \$450/unit = \$134K

Labor, 168 hrs x 3 laborers x \$35/hr = \$18K

Replacement power @ 2 cents/k Wh

168 hrs x 250,000 kW x 0.02 = \$840K

Total cost for the event \$992K

Thus it can be seen that if the use of the more robust and durable CFCC candles were to reduce even one replacement event/year the savings would be significant even if the premium for the CFCC candle were to double.

Commercialization and Marketing Plan

High temperature gas filtration provides an ideal opportunity for implementation of CFCC elements. In this basic format, implementation will occur simply by substituting CFCC filters for existing filters in Westinghouse Strategic Facilities and related DOE Clean Coal Demonstration programs. Since DLC will be a commercial supplier of CFCC elements, CFCC elements will be available to all users.

The proposed commercialization path will consist of: (a) Demonstrate superior filters in this program, (b) Establish a source of supply: DuPont Lanxide Composites, (c) Substitute CFCC filters in available Westinghouse strategic test facilities and DOE Clean Coal Technology Demonstration Programs, (d) Provide commercially available filter elements to a global market: DLC, (e) Expand availability of CFCC filters by first meeting "high-end" highly leveraged cost benefit objectives to be realized by utilities in coal-fired IGCC and APFBC applications. Then cost reduce filters to meet high-volume industrial/commercial markets.

Solar Turbines for Industrial Gas Turbines

The objective of the program is to provide DuPont Lanxide Composites Incorporated with an assessment of the use of their Nicalon™ reinforced alumina matrix composite in selected gas turbine applications. This feedback is intended to enhance and expedite DuPont Lanxide's ability to develop and commercialize their continuous fiber reinforced ceramic matrix composites.

The use of DuPont Lanxide's DIMOX™ directed metal oxidation process Nicalon™ reinforced alumina composite in three combustor components and a first stage nozzle was studied. An impact study was completed in terms of performance, energy savings, economic benefits, strategic factors, and environmental effects. Each component application was defined, and preliminary stress analysis was performed. The conclusions from this work are as follows:

- The use of ceramics in a gas turbine hot section allows an increase in the Total Rotor Inlet Temperature (TRIT) and corresponding increases in electrical output and electrical thermal efficiency.
- Reliability, Availability, and Maintainability are important buying criteria in the 1990's and will continue to be important after 2000. In the future, efficiency and emissions will become more important than first cost.
- Due to their greater efficiency, ceramic hot section gas turbines would use only 83% of the natural gas that would be required by metallic hot section gas turbines to produce the same amount of energy, giving a savings of 17%.

- Ceramic hot section gas turbines create significantly less CO₂ and NO_x than metallic hot section gas turbines.
- CFCCs do not use significant quantities of strategic elements.
- The use of the DuPont Lanxide DIMOX™ Nicalon™ reinforced alumina composite is not recommended in the first stage nozzle at this time. Extensive redesign and improvement in material properties will be required before this CFCC can be used for the nozzle application.
- The definition and stress analysis have shown that the DuPont Lanxide DIMOX™ Nicalon™ reinforced alumina composite is a viable candidate for the combustor hot wall applications.

Foster Wheeler Development Corporation

The objective of this program is to assess the potential of DuPont Lanxide Composites developed CFCC material in industrial applications. This includes applications analyses, definitions of performance requirements, targeted material properties, systems analyses, conceptual design studies and a commercialization plan.

Industrial Applications: These included high temperature heat-exchangers; air preheaters; steam/methane reformers; and applications including tube supports for high temperature fired heaters, high temperature valves and structural division walls. After careful consideration of market potential, energy savings, environmental benefits, technical feasibility and potential job creation, the high temperature heat exchanger and steam/methane reformer were selected for the study. An advanced combined-cycle plant incorporating a CFCC heat exchanger would allow an increase in plant efficiency

from 35% to 47%. Such a plant will generate 35% more power per ton of coal. It was also estimated that CFCC steam reformer tubes have the potential to save 19% in feed stock requirements and 30% in energy requirements.

The efficiency of a conventional coal-fired power plant is approximately 35%. With the continued development of high-temperature gas turbines, it is possible to substantially improve today's power plant efficiencies by means of a high-temperature, combined Brayton/Rankine cycle, in which the conventional steam cycle is topped with a Brayton cycle using an externally-fired gas turbine.

Design Studies of CFCC Heat Exchanger: Four combined-cycle systems are being considered to achieve higher conversion efficiency. These are the Integrated Coal Gasification Combined Cycles (IGCC); Second Generation Pressurized Fluidized Bed (SGPFB); Externally Fired Combined cycle EFCC); and High Performance Power Systems(HIPPS). Among these power cycles the EFCC and the HIPPS are candidates for a CFCC test exchanger. The HIPPS cycle was selected as it offers higher generating efficiency and minimizes problems with high temperature corrosion.

The CFCC heat exchanger is designed for heating air from 1400F to 1800F in a High Temperature Advanced Furnace (HITAF) as part of the 290 - MWe HIPPS plant. The flue gas entering the heat exchanger is at 2935F and is sufficiently clean to prevent ash deposition and corrosion problems. The proposed heat exchanger design is a multipass shell-and -tube construction and incorporates a counter flow design.

Design Studies of CFCC Steam Reformer: The steam reforming of natural gas to produce a mixture of hydrogen and carbon monoxide is a process which is practiced nationwide by chemical and steel manufacturers and oil refiners. The steam reforming

reaction is carried out at relatively high temperatures and pressures. Since the maximum pressure and temperature for steam reforming are limited by material considerations, the product gas is further compressed downstream of the reformer to meet process requirements. This compression cost is a major factor in determining the economics and energy consumption of these processes. A more energy-efficient steam reforming reaction would be possible, if stronger, more durable and heat resistant materials such as the CFCCs were available for certain components of reforming furnace.

In order to fully take advantage of the high-temperature properties of the CFCC material a CFCC reformer in a hydrogen plant that would produce high-pressure hydrogen without using a downstream compressor was chosen. A standard hydrogen plant using a conventional metallic steam reformer and producing 50-MM SCFD hydrogen product gas at 351 psig was used as reference. Since most downstream processing options in a hydrogen plant require product gas at higher pressures, system design of 50-MM SCFD hydrogen plant delivering product gas at 940 psig was considered for a CFCC reformer application.

For the high-pressure plant, it was determined that a two-stage reformer will best utilize the high-temperature strength of the CFCC material. The first stage will use metallic tubes and the second stage CFCC tubes yielding a final outlet synthesis gas at 1000 psig and 1815F. A two-cell reformer, which arranges the flow and duty to be equally split between two cells, was chosen.

Commercialization Plan: An outline of a commercialization plan was also developed. This involves (a) Technical Development (conceptual design, small-scale and full-scale tests, intermediate and full-scale (100 MWe) demos and development of codes

and standards; (b) Utility awareness and perception and (c) Commercial plant cost and market penetration studies.

Performance Requirements, Technical and Economic Targets: In these tasks the technical performance requirements, targeted material properties and economic targets were defined.

Laboratory Exposure Tests: Several dozen CFCC coupons provided by DuPont Lanxide Composites were exposed to coal gas and steam reformer environments for 500, 1000, 1500 hrs. under ambient pressures. After exposure, the coupons were tested for strength and weight change. Microstructural studies of exposed specimens were also conducted. These studies indicated no significant corrosion or degradation of properties.

General Electric Power Generation

The use of ceramics in industrial turbomachinery applications will provide substantial pay off for components which require the use of chargeable cooling air. "Chargeable" cooling air is cooling air from the turbine compressor that is used to actually cool components aft of the first stage turbine nozzle and therefore has a direct negative impact on engine cycle efficiency. The first stage turbine buckets and shrouds currently require chargeable cooling air. Other components which would yield significant payoffs are the combustors, transition piece and the first stage turbine nozzle. Currently, all of these components are manufactured from superalloys which require aggressive air cooling to maintain metal temperature below 1700F. The use of ceramics which are capable of operation at 2200F or higher will result in a greatly reduced amount of chargeable cooling air to the gas turbine.

The reduction or elimination of the cooling air currently required for metallic components will allow more of the air discharged from the compressor to be available to do work in the turbine, resulting in greater performance and efficiency. It is estimated that for a 160 MW gas turbine stage 1 shroud, a 1% decrease in chargeable cooling air would increase the power output of the gas turbine by 1% which would result in fuel savings of approximately \$500,000 per machine per year.

The use of a ceramic composite combustion liner can have a two fold benefit, emissions reduction and fuel savings through cycle efficiency improvements. The reduction in emissions will result from the elimination of film cooling. Removing the film cooling will increase the temperature on the liner walls, allowing for complete combustion to occur. This will reduce carbon monoxide (CO) and unburned hydrocarbons (UHC). Also, the compressor discharge air that was used for film cooling now can be introduced through the head-end of the combustion liner to aid in the reduction of oxides of nitrogen (NOx) formation by premix combustion techniques.

CFCC components must meet significantly more demanding material requirements for an industrial gas turbine (IGT) application relative to previously identified applications, such as aircraft engine exhaust flaps. IGT design considerations for CFCC's include the requirements that ceramic matrix composites remain in service for long periods of times at elevated temperatures and can withstand the thermal shock of sudden machine off-load conditions or trips. Interface coatings (between fiber and matrix) are currently the subject of significant investigation due to the concern of strength degradation in the temperature range of interest (1500F to 2200F). Ceramic matrix composite/metal joints or attachments also present a unique challenge relative to existing experience with metal to metal interfaces. Component design and

development, in general, has been hampered by lack of material property data, limited materials availability, limited supplier experience with fabrication techniques, and the high cost of these experimental materials.

In Phase I specific requirements of the shrouds and combustors were defined and related to the CFCC systems and capabilities. Studies were performed to define preliminary designs of the CFCC turbine shroud and combustion liner. The shroud and combustion liner components studied were for a MS0017FA (7FA) retrofit application, a 160 MW land-based IGT. GE Power Generation Engineering evaluated five material systems for the shroud application: DuPont Lanxide Composites (DLC) DIMOX® directed metal oxidation process material, DuPont Lanxide Composites CVI, GE CR&D/ Textron, Amercom, and Babcock & Wilcox (B&W). Due to limited funds, GEPG down selected to 3 of the 5 systems for the combustor application: DLC DIMOX®, DLC CVI, and GE CR&D/Textron. The team visited all 5 vendors to discuss the IGT applications, material property needs and tour the manufacturing facilities. Monthly updates were provided to the DOE through GE CR&D's monthly reports.

Operating and design requirements for the ceramic components were identified based on the current metallic part requirements. Configuration requirements for each component were also driven by the desire to integrate the CFCC component design into the current 7FA turbine and combustion system.

A material database survey was conducted. All five material suppliers were asked to provide property data in a standardized format at four different temperatures: 70F, 1000F, 2200F, and 2500F. The data were characterized as: 3-test data, 2-engineering estimate, 1-micromechanical estimate and blank-no data provided.

Preliminary designs were generated, incorporating fabrication information gathered from the vendors list. Steady state thermal and stress analyses were conducted for each component. Material limiting locations for each configuration were identified, based on stress and temperature distributions. The quality of the material property was reviewed and the required action identified to meet the stress in the limiting locations.

A matrix based decision process was devised to evaluate the five material systems. The focus of the material evaluation process was to identify the material systems that are capable of meeting CFCC program needs for near and mid-range applications. Four major categories were identified: design, material properties, manufacturing, and cost. Based on these four categories the DLC DIMOX® material system received the most favorable placing.

It should be made clear, the composite system evaluated in this report are not at the same level of maturity. Given the difficulty to predict future material advancements for each of the suppliers, the evaluation team focused on "today's status" of the material system development.

It was concluded, based on engineering judgment, that all of the material systems could be made to work. Currently, there does not exist enough information to screen-out any of the materials evaluated. Long term material property data is needed.

2.0 THE DIMOXTM DIRECTED METAL OXIDATION PROCESS

The DIMOXTM directed metal oxidation process is utilized to fabricate continuous fiber ceramic matrix composites with an alumina matrix. The alumina matrix forms by a chemical oxidation reaction of a molten aluminum alloy with oxygen and “grows” into the spaces between individual fibers and the fiber bundles to form a fully dense ceramic matrix composite. The process sequence for a fiber reinforced alumina matrix composite is shown in Figure 1.

Figure 1. Process sequence for a fiber reinforced alumina matrix composite fabricated via the DIMOXTM directed metal oxidation process.

Prior to matrix growth, a porous fiber preform is fabricated by conventional preforming techniques such as fabric lay-up, filament winding, braiding, weaving, etc. The porous fiber preform is then placed into a chemical vapor infiltration (CVI) reactor, where a duplex coating of boron nitride (BN) and silicon carbide (SiC) is deposited on all the fibers. The purpose of the BN coating is to provide a weak interface on the surface of the fibers, which is vital to the mechanical performance characteristics of the composite. This weak interface provides a path for crack deflection and fiber pull-out which is required for high strength, high fracture toughness, and graceful failure. The purpose of the SiC coating is twofold. First, it must protect the BN interface coating and NicalonTM fibers from attack by the molten aluminum during the DIMOXTM directed metal oxidation process. If either of these reactions are extensive, the weak interface will not be maintained and a significant fraction of the continuous fibers may be damaged. Damage to the interface and fibers results in a degradation of the mechanical properties of the composite.

After CVI coating, the preform is placed in contact with an aluminum alloy (either solid or molten) inside a refractory container. The process is illustrated schematically in Figure 2. When heated in air above the melting point of the aluminum alloy, a highly interconnected ceramic matrix phase of alpha alumina "grows" into the pores of the preform starting from the alloy/preform interface. Present in this reaction product matrix is an interconnected network of microscopic aluminum channels. The aluminum alloy, driven by surface energy forces, wicks from the molten reservoir through the microchannels to the growth front. There, the alloy continues to react with oxygen and sustain the alumina matrix growth process. Growth rates depend on the process conditions and the desired microstructural characteristics of the matrix.

Typical growth times require from 50 to 200 hours at the growth temperature for components with .1 to .3" in thickness.

Figure 2. Schematic of DIMOX® directed metal oxidation process for alumin matrix composites.

The growing matrix fills the large spaces between fiber bundles and the small spaces between individual fibers extremely well, leading to an as-grown composite with less than 2 vol. % porosity. The as-grown composite contains approximately 10 vol. % of the parent aluminum alloy in the network of interconnected microscopic channels. For most high temperature applications, the presence of interconnected aluminum in the composite is undesirable. Therefore, the residual aluminum is extracted from the interconnected channels. The process for effectively removing the residual aluminum

from the alumina matrix is considered proprietary and will not be discussed in this report. After aluminum metal removal, the composite contains 5-10 vol.% interconnected porosity (on the order of 1-2 μm diameter) and less than 2 vol. % residual metal in the form of closed pockets.

The directed metal oxidation process allows for the fabrication of components to net or near net shape by coating all preform surfaces except the one in contact with the alloy with a gas permeable barrier layer (see Figure 2). This growth barrier brings the growth process locally to a halt when the growth front has traversed the preform and comes into contact with the barrier. At that time, the barrier material reacts with the aluminum alloy to form a reaction product that prevents further growth. Hence, simple flat plates are fabricated by coating the sides and top of the preform with a barrier. Tubular components are often fabricated by inside out growth, i.e., alloy is contained inside the preform and the matrix grows in a radial direction to the outer surface which is coated with the barrier compounds. With variations of this technique, a wide range of configurations can be fabricated. If necessary, components may then be machined to final dimensions. This usually involves minor trimming of edges and/or grinding and polishing of surfaces.

3.0 PROPERTIES OF THE NICALON™/ALUMINA COMPOSITE

Process feasibility had been demonstrated on 1-D, 2-D and 3-D Nicalon™ fiber reinforced alumina matrix composites prior to Phase I of the DOE-CFCC program. Commercialization of the Nicalon™/Alumina composite fabricated with layers of harness satin weave (HSW) fabric in a 2-D, 0/90° lay-up was already in progress. Preforming techniques were well established and had been used on a variety of shaped components including plates, cylinders, and more complicated hardware. The BN/SiC interface coatings were developed and provided a weak fiber/matrix interface and therefore good fracture toughness and strength. Matrix growth via the DIMOX™ directed metal oxidation process resulted in full densification of the fiber preform with minimum damage to the interface coatings or fiber reinforcement. Representative components and prototypes of various geometries had been produced and tested. The mechanical properties of the composite had been measured and reported in the open literature. A brief summary of the material properties is given below. (Note: The material properties discussed below are for a 2-D, 0/90°, 12 HSW Nicalon™/Alumina composite with 35-40 vol. % fibers and all of the interconnected residual aluminum removed. All samples were tested in the as-machined condition without any externally applied oxidation protection coating.)

Four point flexural strengths of the Nicalon™/Alumina composite are presented in Table 1, and range from 461MPa at 23°C to 340 MPa at 1400°C. The 23°C strength is retained at 1200°C for short term exposures (<15 min.). The gradual decrease in flexural strength with further increase in test temperature is attributed to degradation of the Nicalon™ fiber.

Table 1. Flexural strength of the Nicalon™/Alumina composite.

Test Temp. (°C)	Number of Samples Tested	Average Strength (MPa)	Maximum Strength (MPa)	Minimum Strength (MPa)
23	8	461 ± 28	511	438
1200	10	488 ± 22	517	440
1300	4	400 ± 12	412	386
1400	4	340 ± 11	348	325

The tensile strength of the Nicalon™/Alumina composite follows the same trend. As shown in Table 2, the 23°C tensile strength of 260 MPa is retained up to 1200°C in air. The average tensile strength at 1370°C is 230 MPa. The small drop in tensile strength at temperatures above 1200°C is attributed to degradation of Nicalon™ fiber.

Table 2. Tensile strength of the Nicalon™/Alumina composite.

Test Temp. (°C)	Number of Samples Tested	Average Strength (MPa) Mean ± St. Dev.	Proportional Limit (MPa)	Young Modulus (GPa)
23	3	264 ± 9	42	140
1100	3	252 ± 26	63	—
1200	3	253 ± 52	54	—
1300	3	225 ± 6	68	—

The relatively high strength of the NicalonTM fiber in combination with the weak fiber-matrix interface results in high composite fracture toughness. The fracture toughness of the NicalonTM/Alumina composite is in the range of 28 to 16 MPa√m between 23 and 1400°C respectively. As observed in Table 3, 84% of the 23°C fracture toughness is retained at 1200°C.

Table 3. Fracture toughness of the NicalonTM/Alumina composite.

Test Temp. (°C)	Number of Samples Tested	Average Fracture Toughness (MPa√m)
23	3	27.8 ± 4.6
1200	3	23.3 ± 2.7
1300	3	19.2 ± 2.8
1400	3	15.6 ± 3.7

To demonstrate the oxidation resistance of the NicalonTM/Alumina composite, tensile stress rupture tests were conducted. Tests consisted of exposing samples to elevated temperatures (1000-1200°C) in air for extended times under the application of a constant load which was periodically increased. The applied tensile stress is plotted versus time as shown in Figure 3. The end of each plot indicates the highest applied stress and the time when the sample failed. Over the duration of the tests, exposures of over 500 hours at 1100°C and over 1000 hours at 1200°C were achieved with an applied tensile stress in the range of 65-100 MPa. All of the samples failed when the applied stress was increased to 100 MPa.

Figure 3. Tensile stress rupture behavior of the Nicalon™/Alumina composite.

The low cycle tension-tension fatigue behavior of the Nicalon™/Alumina composite was evaluated at 23, 1000, 1100, and 1370°C in air. The results are summarized in Table 4. Note that the test protocol was not always the same for all of the data presented in Table 4. Therefore, direct comparisons are not always possible. Nevertheless, several important points are noteworthy. Over two million cycles were completed without failure at 1000°C with an applied tensile stress between 8 and 83 MPa at a frequency of 5 Hz. Residual tensile strengths at temperature were measured for samples exhibiting no failure following 10,000 cycles at 1100 or 1370°C in air. Over 50% of the elevated temperature tensile strength was retained for these samples. The S-N plot for the 1100°C tests is shown in Figure 4, suggesting a tendency towards an endurance limit of ~80 MPa at 1100°C in air.

Table 4. Fatigue data for the NicalonTM/Alumina composite.

Test Temperature	Maximum Stress (MPa)	Frequency (Hz)	Cycles to Failure	Residual Strength at Temp. (MPa)
23°C	72	1	172,912	
	72	1	235,858	
	138	1	150,956*	
	138	1	166,459*	
	166	1	351,600	
	166	10	118,360	
1000°C	83	5	2,195,000*	
	103	0.3	142,577*	
	103	5	117,843	
	103	5	175,620	
	124	1	27,712	
	124	5	66,676	
	138	5	10,508	
1100°C	60	1	10,000*	185 149
	120	1	10,000*	
	180	1	603	
	200	.25	0	
	150	.25	12	
	120	.25	1,718	
	100	.25	2,106	
	90	.25	33,838	
	80	.25	100,000*	
1370°C	58	1	10,000*	153
	115	1	2,480	
	173	1	128	

*Test stopped prior to failure

Figure 4. Low cycle fatigue behavior of the Nicalon™/Alumina composite.

Additional material properties measured on the Nicalon™/Alumina composite are summarized below in Table 5.

Table 5. Material properties of the Nicalon™/Alumina composite.

Property	Units	23°C	1000°C	1200°C
Density	g/cm ³	2.9	—	—
Coefficient of Thermal Expansion	ppm/°C	5.8	5.2	—
Through Thickness Thermal Conductivity	W/m•K	8.7	5.7	5.5

4.0 PHASE I, TASK 3.1, SCOPE OF WORK

The NicalonTM/Alumina composite fabricated via the DIMOXTM directed metal oxidation process combines light weight, high strength, high fracture toughness, and high temperature environmental stability, making it an excellent candidate for the industrial applications targeted for the commercialization of CFCC materials. Prior to Phase I of the DOE-CFCC Program, process feasibility on the NicalonTM/Alumina composite had been demonstrated, a material property data base been generated, and commercialization efforts were already underway. However, several process improvements and performance enhancements were identified as critical to the successful wide-spread industrial commercialization of the NicalonTM/Alumina composite. Among them were:

- a safe, scaleable, CVI process for the SiC fiber coatings
- decreased matrix microcracking
- increased matrix microcracking yield stress
- increased thermal conductivity
- increased high temperature strength
- extended elevated temperature service life
- reduced cost of fiber preforms

DuPont Lanxide Composites scope of work for the Phase I, Task 3.1 effort focused on potential process and performance improvements to the currently available NicalonTM/Alumina composite. Specifically, five materials development tasks were conducted which demonstrated process feasibility on five different CFCC material systems. Each of the five materials development tasks addressed one or more of the

development needs outlined above. A brief description of these five materials development tasks and the rationale for their selection and development is given below. The results are presented in Section 5.0. (Note: The results of Task 3.1 are presented here in order of importance and level of effort. Therefore, specific task numbers per the original statement of work are not described in sequence.)

In addition to the materials development tasks outlined below for Task 3.1, the existing material property data base on the currently available NicalonTM/Alumina composite was provided to the OEM partners conducting Task 1.0, Applications Assessment (i.e., Foster Wheeler, General Electric, Solar Turbines and Westinghouse). The results of their applications assessments are presented under separate cover.

Task 3.1.5 An improved SiC-CVI Process for the NicalonTM/Alumina Composite

The CVI process developed in the laboratory for coating BN-coated NicalonTM fibers with a protective SiC overcoat was deemed unsafe for scale-up to a larger CVI reactor. This conclusion was based on the observation that potentially dangerous quantities of pyrophoric compounds were deposited in the downstream plumbing of the larger reactor. This necessitated the development of a new set of SiC-CVI process conditions to enable process scale-up at a manufacturing scale. This new set of SiC-CVI process conditions was established during in-house experimental work at DuPont Lanxide Composites (outside the scope of the CFCC program), and was demonstrated to be safe and scaleable to a manufacturing scale CVI reactor. The purpose of Task 3.1.5 of this program was to evaluate this new set of SiC-CVI process conditions for its utility in the NicalonTM/Alumina composite system. During Phase I, Task 3.1.5, DuPont Lanxide Composites evaluated the suitability of this new CVI process for producing protective SiC coatings, and evaluated the mechanical behavior of

the NicalonTM/Alumina composites that were fabricated. The overall objective was to produce the NicalonTM/Alumina composite utilizing safe, scaleable, manufacturing processes and with mechanical performance characteristics equal to or better than the currently available NicalonTM/Alumina composite, which utilized the laboratory CVI process for SiC coatings.

Task 3.1.3 NicalonTM Fiber + SiC Particulate Reinforced Alumina

In the NicalonTM/Alumina composite, the mismatch in thermal expansion between the NicalonTM fiber bundles (~4.0 ppm/°C) and the alumina matrix (~8.0 ppm/°C) causes microcracking in the matrix-rich regions of the composite when it is cooled from elevated temperatures. It is believed that the pressure of microcracks in the matrix-rich regions of the composite results in a relatively low proportional limit, commonly referred to in CFCCs as the matrix microcrack yield strength. Furthermore, the microcracks in the matrix of the composite provide paths for oxygen ingress and thus the potential for high temperature oxidation of the interface coatings and/or fibers. One potential approach to minimize matrix microcracking would be to add SiC particulates to the matrix-rich region of the composite during fabrication. The SiC particulate additions would decrease the overall thermal expansion characteristics of the matrix-rich regions thus minimizing the overall thermal expansion mismatch between fiber bundles and the matrix-rich regions. Furthermore, the addition of a higher thermal conductivity phase (SiC particulates) could increase the overall thermal conductivity of the composite. During Phase I, Task 3.1.3, DuPont Lanxide Composites evaluated the processing, thermal, oxidation, and mechanical property characteristics of a hybrid NicalonTM/Alumina composite which had SiC particulates added to the matrix-rich regions of the composite. The overall objective was to demonstrate process feasibility on a hybrid NicalonTM/Alumina composite with reduced matrix

microcracking, increased microcrack yield strength, improved oxidation resistance, and increased thermal conductivity over the currently available NicalonTM/Alumina composite which does not contain SiC particulate additions.

Task 3.1.2 HPZTM Fiber Reinforced Alumina

The high temperature strength of the NicalonTM/Alumina composite begins to decrease at temperatures above about 1100°C due to degradation of the NicalonTM fibers. One potential approach to increase the high temperature strength of the composite would be to replace the NicalonTM fiber with another fiber with better high temperature strength. The HPZTM fiber from Dow Corning was considered the leading candidate and selected for evaluation. During Phase I, Task 3.1.2, DuPont Lanxide Composites evaluated the processing and mechanical property characteristics of an HPZTM fiber reinforced alumina matrix composite. The overall objective was to demonstrate process feasibility on a new CFCC material system with increased high temperature strength over the currently available NicalonTM/Alumina composite.

Task 3.1.4 AlmaxTM Fiber Reinforced Alumina

A second approach to minimize matrix microcracking and increase the microcrack yield strength would be to replace the NicalonTM fiber with an alumina fiber having a coefficient of thermal expansion much closer to that of the alumina matrix. An alumina fiber might also offer improved high temperature oxidation and corrosion resistance. The AlmaxTM high purity alumina fiber from Mitsui Mining was considered the leading candidate and selected for evaluation. During Phase I, Task 3.1.4, DuPont Lanxide Composites evaluated the processing and mechanical property characteristics of an AlmaxTM fiber reinforced alumina matrix composite. The overall objective was to demonstrate process feasibility on a new CFCC material system with reduced matrix

microcracking, increased microcrack yield strength, and improved oxidation resistance over the currently available NicalonTM/Alumina composite.

Task 3.1.1 Chopped NicalonTM Fiber Reinforced Alumina

One potential approach to make cost effective complex shaped fiber preforms is to use chopped rather than continuous NicalonTM fibers. During Phase I, Task 3.1.1, DuPont Lanxide Composites evaluated the processing and mechanical property characteristics of a chopped NicalonTM fiber reinforced alumina matrix composite. Commercially available chopped NicalonTM fiber mats were used as preforms to fabricate flat plates for characterization. Initial studies were directed toward optimizing the volume fraction of the chopped fibers and varying the CVI process conditions to obtain the desired interface coating thickness. The overall objective was to demonstrate process feasibility on a chopped NicalonTM/Alumina composite and to determine if the mechanical property characteristics warranted further preform development and optimization.

5.0 RESULTS OF TASK 3.1, PROCESS FEASIBILITY

5.1 Nicalon Fiber Reinforced Alumina (Task 3.1.5)

Processing of Nicalon™/Alumina Composites:

Flat plate preforms of continuous Nicalon™ fiber were fabricated from as-purchased Nicalon™ fabric. The Nicalon™ fabric was of a 2-dimensional, 0°/90° orientation, 12 harness satin weave (HSW) construction. One layer or "ply" of this fabric measures ~.015" thick. The flat plate preforms were constructed by stacking 8 plies of the 12 HSW fabric by hand. The resulting preform measured approximately 1/8" thick. The fabric plies were cut to 6 x 9" prior to lay-up.

The Nicalon™ preforms were then placed in a CVI reactor and first coated with a thin layer of boron nitride (BN) and then coated with a thicker layer of silicon carbide (SiC). The thickness of each CVI layer was determined by the specific CVI process conditions that were utilized and the amount of time that the preform was held in the CVI reactor.

After the preform was CVI coated with BN and SiC, it was densified via the DIMOX™ directed metal oxidation process, as described in Section 2.0 of this report. The resulting composite consisted of BN/SiC coated Nicalon™ fibers in a matrix of alumina which contained some residual aluminum.

After matrix growth, the NicalonTM/Alumina composite was subjected to a metal removal process which effectively removed the residual aluminum from the interconnected channels within the alumina matrix. The metal removal process resulted in 5-10 vol. % of very fine interconnected porosity within the alumina matrix.

The important constituents of the NicalonTM/Alumina composite are the continuous NicalonTM fibers, the BN debond layer, the protective SiC overcoat layer, the alumina matrix, and the interconnected porosity. The mechanical properties of the composite are dominated by the mechanical properties of the continuous NicalonTM fibers. However, a good BN debond layer is also essential because it provides a weak interface at the fiber surface. A weak interface is required for crack deflection and fiber pull-out, which imparts good strength and fracture toughness. The main function of the SiC overcoat layer is to protect both the BN debond layer and the NicalonTM fibers from oxidation and/or attack during the growth of the alumina matrix. If a protective SiC coating of the appropriate thickness is not covering the BN coated NicalonTM fibers, the characteristic weak interface will not be maintained, and a significant fraction of the load-bearing continuous NicalonTM fibers may be damaged. This will result in a significant degradation of the composites' mechanical properties. Therefore, it is essential to the quality of the NicalonTM/Alumina composite that the BN coated NicalonTM fibers are covered with a protective layer of SiC. The methodology used to determine the utility of the new SiC-CVI process developed for the fabrication of the NicalonTM/Alumina composite on a manufacturing scale is described below. The same methodology was also used to evaluate the four other CFCC materials developed during Phase I, Task 3.1, and discussed later in this report.

Methodology for Composite Evaluation:

After fabrication, the NicalonTM/Alumina composite plates were surface ground, cross-sectioned for metallographic preparation, and machined into test coupons for mechanical testing. Six characterization techniques were utilized to evaluate the quality and performance characteristics of the composites produced. They were:

1. Optical microscope evaluation of the composite microstructure
2. Flexure testing at 23 and 1200°C
3. Tensile testing at 23, 1000 and 1200°C
4. Tensile stress rupture testing at 1100°C
5. Low cycle fatigue testing at 1100°C
6. Scanning electron microscope evaluation of fracture surfaces

Microstructural evaluation was used to verify that the SiC coatings were protective during matrix growth, and to measure the thickness of the BN and SiC coatings. The target thickness for the BN debond layer is between .5 and 1.0 μm throughout the 8-ply composite. The target thickness for the SiC overcoat layer is between 3 and 5 μm throughout the 8-ply composite.

Typically, the SiC coatings are not uniform throughout the cross-section of the 8-ply composite, i.e., they are thickest at the outer plies and thinnest at the inner plies. The targeted 3 to 5 μm SiC coating thickness is considered the minimum coating thickness required to provide adequate protection and acceptable mechanical performance. (The variations in SiC coating thickness will be discussed in greater detail later in this report.) If the composite microstructure indicated that the coatings

and fibers were oxidized and/or attacked during matrix growth, the material was considered to be unacceptable and no further characterization work was performed. When the SiC coatings provided adequate protection for the BN interface and NicalonTM fibers, the composite microstructure was considered acceptable and the mechanical properties were characterized.

Flexure testing is an efficient and economical approach for evaluating the quality of a ceramic matrix composite. The flexure strength of the NicalonTM/Alumina composite is mainly determined by the volume fraction of continuous NicalonTM fibers. Therefore, 23 and 1200°C flexure testing was conducted as a screening test to verify that the NicalonTM/Alumina composite had the appropriate volume fraction of NicalonTM fibers and that the fibers and BN debond layer were protected during matrix growth. For the 2-D, 0°/90°, 8-ply, 12 HSW NicalonTM/Alumina composite, the targeted volume fraction of NicalonTM fibers is 35 vol. %. A NicalonTM/Alumina composite with 35 vol. % fiber have typically exhibited flexural strengths of approximately 400 ± 25 MPa. Furthermore, if the SiC coatings are of the appropriate thickness and protective, the 1200°C flexural strength is comparable to that at 23°C. Target values of 375 ± 25 MPa at 1200°C have been established. If the NicalonTM/Alumina composite exhibited 23 and 1200°C flexure strengths of 400 ± 25 and 375 ± 25 MPa, respectively, the composite was then evaluated under axial tensile loads at 23°C.

The flexure test is an effective screening technique to evaluate the quality of the NicalonTM/Alumina composite. However, the flexure test data reveal very little about the actual stress-strain behavior of the composite, and are therefore not very useful to designers. Hence, room temperature tensile testing of 6" long, flat, dog-bone shaped tensile coupons was conducted to determine the stress-strain characteristics of the

NicalonTM/Alumina composite. The key properties obtained from the 23°C tensile test, and the targeted values for the NicalonTM/Alumina composite are presented below:

1. Ultimate Tensile Strength (U.T.S. = 250 ± 25 MPa)
2. Proportional Limit (P.L. = 50 ± 10 MPa)
(for CFCC's, referred to as matrix microcrack yield strength)
3. Youngs Modulus ($E = 120 \pm 20$ GPa)
4. Strain-To-Failure ($e_f = .6 \pm .2\%$)

If the composite exhibited acceptable tensile stress-strain behavior at room temperature, then high temperature tensile, tensile stress rupture and low cycle fatigue tests were conducted to further evaluate the composites performance characteristics. However, these tests are more expensive to conduct, and were only performed after thorough process optimization had been demonstrated.

After the appropriate mechanical testing was completed, representative fracture surfaces were examined on the scanning electron microscope (SEM). The objective here was to characterize the mode of fracture and the degree of fiber pull-out. The fracture surfaces of all the constituents (fibers, coatings, and matrix) were examined and characterized. A NicalonTM/Alumina composite which exhibited excellent strength and toughness at room and elevated temperatures exhibited a fracture surface with long fiber pull-out at the BN/NicalonTM interface, and a fracture surface morphology on individual fibers characteristic of strong ceramic fibers (i.e., mirror-mist-hackle crack propagation features).

Microstructural Evaluation:

When chemical vapor deposition (CVD) is used to deposit a ceramic coating on a substrate, the coating is uniform over the exposed surface of the substrate, and increases in thickness with increased exposure time. When chemical vapor infiltration (CVI) is used to deposit a ceramic coating on the fibers making up a porous fiber preform, the reactive gases need to simultaneously infiltrate (diffuse) into the preform while depositing on the surface of the individual fibers. The thickness of the CVI coatings that are deposited can vary considerably within different locations of the fiber preform. The variation in CVI coating thickness with location is determined by the reactive gas diffusion rate, its decomposition rate, deposition rate, the specific fiber volume fraction and fiber architecture, etc. For a given fiber volume fraction and fiber architecture, these kinetic relationships are determined by the specific CVI process conditions that are utilized, i.e., pressure, temperature, flow rate, exposure time, reactor hardware configuration, etc.

The CVI process conditions that were originally developed in the laboratory to deposit a SiC protective overcoat on the BN coated NicalonTM fiber preforms were significantly changed to allow for safe process scale-up during the course of this work. As was expected, the variation in the SiC coating thickness throughout the 8-ply, 2-D, 0/90°, 12-HSW NicalonTM fiber preform was also affected by the change in SiC-CVI process conditions. The change in the SiC coating variation that resulted from the change in the CVI process conditions is documented here. The effects that these changes had on the mechanical properties of the NicalonTM/Alumina composite is documented in the next section.

For reporting purposes, the new set of SiC-CVI processing conditions will be referred to as Condition-B. The original set of SiC-CVI processing conditions will be referred to as Condition-A. Specific details about these processing conditions, i.e., pressure, temperature, flow rates, etc., will not be discussed in these reports due to the proprietary nature of the information.

Two 6 x 9" plates were fabricated for initial microstructural evaluation using the new CVI process, Condition-B. Each plate was processed in a 16" diameter CVI reactor for various time intervals. In addition, a third 6 x 9" plate fabricated in 1992 with the laboratory CVI process, Condition-A, was taken from inventory to be used for comparison purposes. Polished cross-sections were prepared from the leading edge (upstream in the CVI reactor) and trailing edge (downstream in the CVI reactor) of each 6 x 9" plate. For each polished cross-section, the thickness of the SiC coating deposited on the fibers of each ply in the 8-ply preform was measured using an optical microscope. The measured SiC coating thickness was then plotted as a function of location in the composite plate, i.e., from the outer surface to the center ply and from the leading edge to the trailing edge. These plots will be used to graphically illustrate the variation in SiC coating thickness with location in the 8-ply composite plate for the two different SiC-CVI process conditions.

Figure 5 shows the variation in SiC coating thickness with location in the 8-ply composite fabricated using CVI Condition-A. Figure 5 illustrates the type of coating thickness variation considered typical for this CVI process. The coatings on the outer surface are thicker than the coatings on the center ply of the preform. In addition, due to reactive gas depletion effects, areas of the fiber preform that were located upstream in the reactive gases (leading edge) exhibited thicker coatings than areas of the fiber

preform that were located downstream in the reactive gases (trailing edge). Specifically, in the leading edge of the composite plate, the fibers on the outer surface of the preform exhibited 30 μm of SiC coating, compared to only 4 μm of SiC coating on the center ply. Similarly, at the trailing edge of the composite plate, the fibers on the outer surface of the preform had 10 μm of SiC coating, whereas only 3 μm of SiC coating was observed on the center ply.

Figure 5. SiC coating thickness vs. location in the 6 x 9" 8-ply NicalonTM/Alumina composite plate.

Figure 6 shows the microstructural variation from the outer ply to the center ply of the leading edge of this composite plate. Similarly, Figure 7 shows the microstructural variation on the center ply from the leading edge to the trailing edge of this composite plate. These microstructural variations are considered typical for the NicalonTM/Alumina composite processed using CVI Condition-A. Furthermore, these variations are considered acceptable for an 8-ply NicalonTM/Alumina composite, based on the extensive mechanical property testing conducted to date. It is important to emphasize again that the main function of the SiC coating is to protect the BN coated NicalonTM fibers from oxidation and/or attack during matrix growth. Note that in all four microstructures presented in Figures 6 and 7, the SiC coating has protected the BN coated NicalonTM fibers. As long as the minimum coating thickness required for protection is maintained throughout the composite, the variation in coating thickness will have little effect on the composites strength and/or toughness. As was described earlier, for the NicalonTM/Alumina composite fabricated using CVI Condition-A, a minimum SiC coating thickness of 3 μm has been established.

(a) outer surface

(b) center ply

Figure 6. Microstructure of the NicalonTM/Alumina composite showing the variation in SiC coating thickness from the outer surface to the center ply of the 6 x 9" 8-ply composite plate. (Plate No. 100192-TAZ, CVI Condition-A).

(a) leading edge

(b) trailing edge

Figure 7. Microstructure of the NicalonTM/Alumina composite showing the variation in SiC coating thickness from the leading edge to the trailing edge of the 6 x 9" 8-ply composite plate. (Plate No. 100192-TAZ, CVI Condition-A).

Figure 8 shows the variation in SiC coating thickness vs. location in the 8-ply composite fabricated using the CVI Condition-B at an exposure time = t_1 . These data indicate that the variation in SiC coating thickness typical of CVI Condition-A has been significantly reduced by using CVI Condition-B. Here, the SiC coating thickness on the leading edge varied from 9 μm on the outer surface down to 4 μm on the center ply. Similarly, the SiC coating thickness on the trailing edge varied from 5 μm on the outer surface down to 2.5 μm on the center ply. Figure 9 shows the microstructural variation from the outer ply to the center ply at the leading edge of this composite plate. Figure 10 shows the microstructural variation on the center ply from the leading edge to the trailing edge of this composite plate. Note that in all four microstructures, the SiC coating has protected the BN coated NicalonTM fibers from oxidation and/or attack. These results indicate that the new SiC-CVI process Condition-B utilized in the larger CVI reactor produced good quality, protective SiC coatings with improved surface-to-center uniformity throughout the 8-ply preforms.

Figure 11 shows the variation in SiC coating thickness for both of the NicalonTM/Alumina composite plates described above, showing a direct comparison of CVI Condition-A to CVI Condition-B. These data indicate that the coating thickness at the center ply of the trailing edge of the composite plate processed using CVI Condition-B was slightly less than the targeted minimum SiC coating thickness of 3 μm . Therefore, a second 6 x 9" composite plate was fabricated using CVI Condition-B, but this time the preform was exposed to the CVI reactive gases for a longer time = $t_2 > t_1$.

Figure 8. SiC coating thickness vs. location in 6 x 9" 8-ply NicalonTM/Alumina composite plate.

(a) outer surface

(b) center ply

Figure 9. Microstructure of the NicalonTM/Alumina composite showing the variation in SiC coating thickness from the outer surface to the center ply of the 6 x 9" 8-ply composite plate. (Plate No. 031793-3, CVI Condition-B).

(a) leading edge

(b) trailing edge

Figure 10. Microstructure of the NicalonTM/Alumina composite showing the variation in SiC coating thickness from the leading edge to the trailing edge of the 6 x 9" 8-ply composite plate. (Plate No. 031793-3, CVI Condition-B).

Figure 11. SiC coating thickness vs. location in 2-6x9" 8-ply Nicalon™/Alumina composite plates.

Figure 12 plots the variation in SiC coating thickness vs. location for this composite plate. As expected, these data indicate that the increased CVI exposure time resulted in an overall increased SiC coating thickness throughout the 8-ply preform. Furthermore, the minimum coating thickness obtained on the center ply of the trailing edge was 4 μm , i.e., above the targeted minimum of 3 μm . Figure 13 shows the microstructural variation in the center ply from the leading edge to the trailing edge of the composite plate. Once again, the SiC coating produced using CVI Condition-B has protected the BN coated Nicalon™ fibers from oxidation and/or attack.

Figure 14 shows the variation in SiC coating thickness vs. location for the two plates processed using CVI Condition-B, directly comparing the results from exposure time t_1 vs. t_2 . Based on these data, it appears that the optimum exposure time for CVI Condition-B is somewhere between time = t_1 and time = t_2 .

Figure 12. SiC coating thickness vs. location in 6x9" 8-ply NicalonTM/Alumina composite plate.

(a) leading edge

(b) trailing edge

Figure 13. Microstructure of the NicalonTM/Alumina composite showing the variation in SiC coating thickness from the leading edge to the trailing edge of 6 x 9" 8-ply composite plate. (Plate No. 062993-1, CVI Condition-B).

Figure 14. SiC coating thickness vs. location in 2-6x9" 8-ply NicalonTM/Alumina composite plates.

Mechanical Property Evaluation:

The 2-6x9x1/8" plates that were processed using CVI Condition-B were machined into flexure and tensile test coupons for mechanical property evaluation. Figure 15 shows how Plate No. 031793-3 was machined into flexure and tensile test coupons, and their relative location in the plate. Similarly, Figure 16 shows how Plate No. 062993-1 was machined into flexure and tensile test coupons, and their relative location in the plate. Plate No. 031793-3 was exposed to CVI Condition-B for time = t1. Plate No.

062993-1 was exposed to CVI Condition-B for time = $t_2 > t_1$. For both composite plates, the odd numbered flexure coupons were tested at 23°C, and the even numbered flexure coupons were tested at 1200°C (15 min. hold in air). All of the tensile coupons from both composite plates were tested at 23°C.

Figure 15. Location of test coupons in 6 x 9" Nicalon™/Alumina plate.

Figure 16. Location of test coupons in 6 x 9" Nicalon™/Alumina plate.

Table 6 presents all of the characterization and mechanical property data obtained on Plate No. 031793-3. Similarly, Table 7 presents all of the characterization and mechanical property data obtained on Plate No. 062993-1. Figures 17 and 18 plot the variation in flexure strengths and tensile properties as a function of location in Plate No. 031793-3. Similarly, Figures 19 and 20 plot the variations in flexure strengths and tensile properties as a function of location in Plate No. 062993-1. Also contained in all four plots is the variation in SiC coating thickness on the center ply from the leading edge to the trailing edge of the composite plate being evaluated.

Table 6. Characterization data from Plate No. 031793-3.

Plate I.D.	Coupon Location	BN (micr.)	SiC (micr.)	Al-carbide (wt. %)	Flex. Str. (MPa) 20 C	Flex. Str. (MPa) 1200 C	Tensile Str. (MPa) 20 C	Prop. Limit (MPa)	Modulus (GPa)	St. to Fail (%)
No. 031793-3 CVI Conditions-B CVI Time = t1	LE									
	1-w			0.0365	419					
	1-m					427				
	1-c			0.0423	453					
	2-w			0.0172		391				
	2-m	<1.0	4.5		466					
	2-c			0.0143		411				
	3.5-m						257	42	127	0.659
	5.5-m						275	45	122	0.726
	7-w				449					
	7-m					376				
	7-c				479					
	8-w					392				
	8-m	<1.0	4		465					
	8-c					381				
	9.5-m						250	37	109	0.661
	11.5-m						231	37	114	0.621
	13-w			0.0236	443					
	13-m					343				
	13-c			0.0204	431					
	14-w			0.0223		306				
	14-m	<1.0	3		427					
	14-c			0.0145		319				
	15.5-m						229	37	114	0.613
	17.5-m						252	35	105	0.703
	19-w				442					
	19-m					262				
	19-c				455					
	20-w					268				
	20-m	<1.0	2.5		440					
	20-c					254				
	21.5-m						247	37	106	0.674
	23.5-m						247	36	112	0.692
	25-w				397					
	25-m					262				
	25-c				459					
	26-w			0.0272		247				
	26-m	<1.0	2.5		455					
	26-c			0.0146		254				
	27.5-m						283	40	107	0.837
	29.5-m						289	39	117	0.763
	31-w			0.0173	404					
	31-m					231				
	31-c			0.0174	486					
	32-w					331				
	32-m	<1.0	2.5		445					
	32-c					383				
	33-w				464					
	33-m					296				
	33-c				470					
	TE									
AVERAGE		<1.0	3.17	0.02	447	323	256	39	113	0.6949
std. dev.					23	64	20	3	7	0.07
(no. of tests)				(12)	(20)	(19)	(10)	(10)	(10)	(10)

Table 7. Characterization data from Plate No. 062993-1.

Plate I.D.	Coupon Location	BN (micr.)	SiC (micr.)	Al-carbide (wt. %)	Flex. Str. (MPa) 20 C	Flex. Str. (MPa) 1200 C	Tensile Str. (MPa) 20 C	Prop. Limit (MPa)	Modulus (GPa)	St. to Fail (%)
No. 062993-1 CVI Conditions-B CVI Time = t2	LE				454					
	1-c					430				
	1-m				403					
	1-w					468				
	2-c				451					
	2-m	<1.0	7			446				
	2-w						267	62	149	0.748
	3.5-m						281	54	132	0.788
	5.5-m				500					
	7-c				480	464				
	7-m					458				
	7-w				455					
	8-c					401				
	8-m	<1.0	5				251	31	146	0.666
	8-w						249	38	143	0.684
	9.5-m				484					
	11.5-m					427				
	13-c	<1.0	5		463					
	13-m									
	13-w									
	14-c									
	14-m									
	14-w					388				
	15.5-m						259	32	142	0.748
	17.5-m						249	33	135	0.685
	19-c				506					
	19-m									
	19-w					407				
	20-c				449					
	20-m	<1.0	4			336				
	20-w						242	36	133	0.644
	21.5-m						244	29	134	0.662
	23.5-m									
	25-c									
	25-m				469					
	25-w					430				
	26-c				469					
	26-m	<1.0	4			389				
	26-w						224	20	131	0.63
	27.5-m						227	54	127	0.592
	29.5-m				443					
	31-c									
	31-m									
	31-w									
	32-c				429					
	32-m	<1.0	4			393				
	32-w				399					
	33-c					417				
	33-m				453					
	33-w									
	TE									
AVERAGE		<1.0	4.83		457	418	249	39	113	0.6847
std. dev.					30	32	17	13	7	0.06
(no. of tests)					(16)	(14)	(10)	(10)	(10)	(10)

Figure 17. Flexural strength and SiC coating thickness vs. location in 6x9" Nicalon™/Alumina composite plate.

Figure 18. Tensile properties and SiC coating thickness vs. location in 6x9" Nicalon™/Alumina composite plate.

Figure 19. Flexural strength and SiC coating thickness vs. location in 6x9" Nicalon™/Alumina composite plate.

Figure 20. Tensile properties and SiC coating thickness vs. location in 6x9" Nicalon™/Alumina composite plate.

The average 23°C flexure strength of Plate No. 031793-3 was 447 ± 23 MPa, and did not vary significantly with location in the plate. The average 23°C tensile strength of Plate No. 031793-3 was 256 ± 20 MPa and also did not vary significantly with location in the plate. These flexure and tensile strength values are equal to or better than the targeted values of 400 ± 25 MPa and 250 ± 25 MPa, respectively. These results indicate that the overall quality of the composite plate was good and that the volume fraction of NicalonTM fiber was above the targeted minimum of 35 vol. % and consistent over the entire plate.

However, the average 1200°C flexure strength of Plate No. 031793-3 was 323 ± 64 MPa, and showed significant variation with location in the plate. In fact, the plot of 1200°C flexure strength vs. location essentially mirrored the plot of SiC coating thickness on the center ply from the leading edge to the trailing edge of the plate. Specifically, the 1200°C flexure strengths were approximately 400 MPa (i.e. above the target value) near the leading edge of the plate where the SiC coating thickness on the center ply was greater than the targeted minimum of 3 μm . However, the 1200°C flexure strengths dropped well below 400 MPa when the SiC coating thickness dropped below the targeted minimum of 3 μm . The mechanism which causes degradation of the 1200°C flexure strength when the SiC coating thickness drops below 3 μm is believed to be associated with localized coating and fiber attack. The data from the leading edge indicate this attack and associated drop in high temperature flexural strength would not have occurred if the SiC coating thickness on the center ply was kept above the targeted minimum of 3 μm . This was verified during the evaluation of Plate No. 062993-1, which was exposed to the CVI reactor for the longer process time = t_2 , and subsequently exhibited thicker SiC coatings.

The average 23°C flexure strength of Plate No. 062993-1 was 457 ± 30 MPa, and did not vary significantly with location in the plate. The average 23°C tensile strength of Plate No. 062993-1 was 249 ± 17 MPa, and did not vary significantly with location in the plate. In addition, the average 1200°C flexural strength of Plate No. 062993-1 was 418 ± 32 MPa and also did not vary significantly with location in the plate (which was not the case for Plate No. 031793-3). This increased 1200°C flexure strength is attributed to the increased SiC coating thickness above the targeted minimum of 3 μm . Again, these results indicate that the overall quality of the composite plate was very good, that the volume fraction of Nicalon™ fiber was above the targeted minimum of 35 vol. % and consistent over the entire plate, that the BN/SiC coated Nicalon™ fibers were sufficiently protected during matrix growth, and that the SiC coating thickness was above the targeted minimum of 3 μm throughout the composite.

Twelve additional 6 x 9" Nicalon™/Alumina composites plates were fabricated for further microstructural evaluation and mechanical testing. The flexural and tensile property data obtained from this lot of composite plates are presented in Table 8.

The mechanical property data presented in Table 8 indicate that the new SiC-CVI process conditions resulted in high quality Nicalon™/Alumina composites with flexure and tensile strengths equal to or better than the target values, provided the minimum SiC coating thickness of 3 μm was maintained. When the SiC coating thickness dropped below the targeted 3 μm minimum, the flexure and tensile strengths dropped below the target values.

Table 8. Nicalon™/Alumina mechanical property data base.

Plate I.D.	SiC Wt. Gain %	SiC Thickness (micron)	Flex. Str. (MPa) 23 c	Flex. Str. (MPa) 1200 C	Tensile Str. (MPa) 23 C	Prop. Limit (MPa) 23 C	Modulus (GPa) 23 C	Str. to Fail. (%) 23 C	Tensile Str. (MPa) 1000 C	Tensile Str. (MPa) 1200 C
031793-3	54	2-5	447 ± 23 (20)	323 ± 64 (19)	256 ± 20 (10)	39 ± 3	113 ± 7	.69 ± .07		
062993-1	80	4-7	457 ± 30 (16)	418 ± 32 (14)	249 ± 17 (10)	39 ± 13	137 ± 7	.68 ± .06		
062993-2	78	4-7	481 ± 54 (15)	356 ± 78 (15)						
062993-3	59	1-4	378 ± 40 (20)	347 ± 45 (18)	225 ± 47 (10)	40 ± 16	103 ± 5	.56 ± .17		
062993-4	58	1-4	381 ± 35 (12)	361 ± 64 (11)						
080393-1*	75	2-7			268 ± 30 (5)	44 ± 11	108 ± 21	.83 ± .12		
080393-2	65	3-6			296 ± 21 (4)	38 ± 2	97 ± 3	.86 ± .07		
081093-1*	79	2-10			198 ± 42 (5)	42 ± 4	121 ± 18	.31 ± .14		
082093-2	64	3-5			290 ± 25 (3)	36 ± 3	132 ± 1	.78 ± .10	222 ± 10 (3)	203 ± 6 (3)
082093-3*	72	2-7	440 ± 38 (16)	437 ± 32 (16)						
082793-1	62	3-5			298 ± 20 (3)	29 ± 5	112 ± 6	.89 ± .01	215 ± 8 (3)	199 ± 2 (3)
082793-2	63	3-5			300 ± 16 (4)	44 ± 5	116 ± 6	.90 ± .10		
091393-1	59	1-4	341 ± 25 (8)	386 ± 36 (7)	182 ± 3 (4)	44 ± 8	120 ± 17	.23 ± .03		
091393-2	59	1-4	291 ± 12 (6)							

* = plate located in vertical position of CVI reactor, SiC coatings showed significant variations with location on plate

The data in Table 8 also indicate that the Nicalon™/Alumina composites processed with the new SiC-CVI conditions exhibited a slight decrease in both the proportional limit (~50 MPa down to ~40 MPa) and modulus (~140 GPa down to ~120 GPa), compared to the original Nicalon™/Alumina composite. The small decrease in modulus has been attributed to the small decrease in the volume fraction of CVI-SiC, which resulted from the more uniform SiC coatings produced with the new SiC-CVI process. The slight decrease in proportional limit is not understood at this time and will be investigated further during Phase II.

The NicalonTM/Alumina composite plates in Table 8 that exhibited SiC coatings greater than 3 μm were evaluated for high temperature tensile, tensile stress rupture, and low cycle fatigue behavior. The results are summarized below.

The tensile properties for the NicalonTM/Alumina composite processed with the new SiC-CVI process are presented in Table 9. The tensile stress-strain curves are shown in Figures 21-24 as a function of test temperature. In general, the composites exhibited excellent tensile strengths, strain to failure, and fiber pull-out at all three test temperatures. The ultimate tensile strength varied from ~300 MPa at 23°C, to 220 MPa at 1000°C, to ~200 MPa at 1200°C. The proportional limit was ~40 MPa and did not vary with test temperature. The modulus was ~120 GPa at 23 and 1000°C, but dropped to ~100 GPa at 1200°C.

Table 9. Tensile properties of the NicalonTM/Alumina composite.

Test Temp. (°C)	Tensile Strength (MPa)	Prop. Limit (MPa)	Youngs Modulus (GPa)	Strain-to-Failure
23	296 $\pm$ 20 (10)	36 $\pm$ 5	120 $\pm$ 10	.86 $\pm$.10
1000	219 $\pm$ 11 (6)	33 $\pm$ 10	121 $\pm$ 5	.54 $\pm$.05
1200	201 $\pm$ 5 (6)	40 $\pm$ 19	103 $\pm$ 12	.78 $\pm$.04

The average 23 and 1000°C tensile data are plotted together in Figure 24 for direct comparison of the room temperature vs. high temperature stress-strain behavior. The stress-strain curves in Figure 24 indicate that even though the proportional limit did not change with increasing test temperature, the tensile behavior of the composite at stresses above the proportional limit was quite different. When tested at 23°C, small

increases in the applied stress above the proportional limit resulted in relatively large plastic strains. By contrast, when tested at 1000°C, small increases in the applied stress above the proportional limit resulted in relatively small plastic strains. It is believed that the change in the composites stress-strain behavior at elevated temperatures may be related to the changes in the residual stress state in the composite resulting from the thermal expansion mismatch between the matrix and the fibers.

Figure 21. Stress-strain behavior of the Nicalon™/Alumina composite at 23°C. (Plate No. 082093-2 and Plate No. 082793-1).

Figure 22. Stress-strain behavior of the Nicalon™/Alumina composite at 1000°C. (Plate No. 082093-2 and Plate No. 082793-1).

Figure 23. Stress-strain behavior of the Nicalon™/Alumina composite at 1200°C. (Plate No. 082093-2 and Plate No. 082793-1).

Figure 24. Stress-strain behavior of the Nicalon™/Alumina composite. (Plate No. 082093-2 and Plate No. 082793-1).

The tensile-stress-rupture behavior of the Nicalon™/Alumina composite processed with the new SiC-CVI process is shown in Figure 25 for direct comparison with the tensile stress rupture behavior of the original Nicalon™/Alumina composite. Here, 1,700 hours of exposure to air at 1100°C was obtained under an applied stress of 80 MPa. The specimen failed when the applied stress was increased to 100 MPa, similar to the original Nicalon™/Alumina composite. These results are very encouraging in that the applied stress of 80 MPa was ~40 MPa higher than the proportional limit of the composite. Even though microcracks have formed and/or propagated through the matrix, the composite has resisted oxidation and/or failure under the applied load. The excellent stress oxidation resistance of the Nicalon™/

Alumina composite is due to an in-situ internal oxidation protection system inherent to this oxide matrix material system. The details of this internal oxidation protection system will not be discussed here due to the proprietary nature of the information.

The low cycle fatigue behavior of the NicalonTM/Alumina composite processed with the new SiC-CVI process is shown in Figure 26 for direct comparison with the fatigue behavior of the original NicalonTM/Alumina composite. Once again, the results are very encouraging and indicate that the fatigue behavior of the NicalonTM/Alumina composite processed with the new SiC-CVI process was equal to or better than the original NicalonTM/Alumina composite. The data in Figure 26 tend to suggest an endurance limit of ~80 MPa at 1100°C.

Figure 25. Tensile stress-rupture behavior of the NicalonTM/Alumina composite.

Figure 26. Low cycle fatigue behavior of the Nicalon™/Alumina composite.

In summary, microstructural evaluation of the Nicalon™/Alumina composites processed in the larger CVI reactor indicated that the SiC coatings produced with the new CVI conditions were much more uniform throughout the 8-ply Nicalon™ preform than those produced with the laboratory CVI process. It was demonstrated that the targeted minimum SiC coating thickness of 3 μm was obtained, and that these SiC coatings provided excellent protection for the BN coated Nicalon™ fibers during oxide matrix growth. The mechanical testing indicated that the new SiC-CVI process conditions resulted in high quality Nicalon™/Alumina composites which exhibited flexural, tensile, tensile stress rupture, and low cycle fatigue behavior equal to or better than the original Nicalon™/Alumina composites processed with the laboratory CVI conditions.

5.2 Nicalon Fiber + SiC Particulate Reinforced Alumina (Task 3.1.3)

There were four main technical objectives for adding SiC particulates to NicalonTM fiber reinforced alumina composites. They were:

1. decrease matrix microcracking
2. increase matrix microcrack yield strength (proportional limit)
3. increase through thickness thermal conductivity
4. improved oxidation resistance.

Initial preforming optimization experiments were conducted on SiC particulate additions to 3-D braided, 2-D braided and 2-D woven NicalonTM fiber preforms. Process feasibility was first demonstrated with a 3-D braided NicalonTM fiber preform. A representative optical micrograph exhibiting a uniform distribution of SiC particulates in a 3-D braided NicalonTM fiber reinforced alumina matrix composite is shown in Figure 27. A through thickness thermal diffusivity (th. cond. = th. diff. x sp. ht. x density) of 0.0785 cm²/s was measured for this composite system at room temperature. This represents a 2X increase in thermal diffusivity relative to the standard 2-D woven NicalonTM/Alumina composite that does not contain SiC particulates.

Silicon carbide particulate additions to the standard 2-D woven NicalonTM/Alumina composite significantly reduced the amount of matrix microcracking and increased the thermal conductivity by ~25%. This hybrid NicalonTM/Alumina composite had 8-10 vol. % of 220 grit particulates and ~31 vol. % of fibers. Figure 28 shows representative microstructures of two NicalonTM/Alumina composites. The standard NicalonTM/Alumina composite without SiC particulate is shown in Figure 28a. The

hybrid NicalonTM/Alumina composite with SiC particulate is shown in Figure 28b. Note the presence of microcracks in the matrix-rich regions of the NicalonTM/Alumina composite without SiC particulate additions. These microcracks were essentially eliminated when the SiC particulate was incorporated into the matrix-rich regions of the composite. Also shown is the through thickness thermal conductivity for the two composites, indicating 20-25% increase at both 23 and 1200°C. Advances in processing techniques doubled the volume fraction of particulates (~20 vol. %) without a significant decrease in fiber volume fraction. A uniform distribution of 20 vol. % particulates through the thickness of the NicalonTM/Alumina composite has been demonstrated and is shown in Figure 29.

Figure 27. Microstructure of 3-D braided NicalonTM/Alumina composite with SiC particulate additions to matrix-rich regions.

(a) thermal conductivity = 8.7 W/m•K @ 23°C
= 5.5 W/m•K @ 1200°C

(b) thermal conductivity = 11.0 W/m•K @ 23°C
= 6.8 W/m•K @ 1200°C

Figure 28. Microstructure of 2-D woven NicalonTM/Alumina composites.
(a = standard NicalonTM/Alumina without SiC particulate)
(b = hybrid NicalonTM/Alumina with 10 vol. % SiC particulate)

Figure 29. Microstructure of 2-D woven NicalonTM/Alumina composite with 20 vol. % SiC particulate additions.

Subsequent activities focused on SiC particulate additions to 2-D woven NicalonTM/Alumina composites only. The 2-D and 3-D braided preforms were omitted from further optimization studies. The technique developed for introducing particulate into 2-D woven fiber architectures was successfully optimized to the stage where reproducible composite microstructures were produced. In addition, the 2-D woven fiber preforms are significantly less expensive than their 2-D and 3-D braided counterparts, and therefore offer a more attractive economic solution to CFCC fabrication.

Room temperature tensile tests were conducted on the hybrid NicalonTM/Alumina composites containing ~10 vol. % of 220 grit SiC particulates. An average tensile strength of 190 ± 15 MPa was observed. The test results also indicated no significant increase in the matrix microcracking yield strength relative to the standard NicalonTM/Alumina composite without particulate additions. Figure 30 shows a typical tensile stress-strain curve for the standard NicalonTM/Alumina composite without SiC particulate additions. This material exhibits a matrix microcracking yield strength of ~50 MPa and an ultimate tensile strength of ~300 MPa. Figure 31 shows a typical tensile stress-strain curve for the hybrid NicalonTM/Alumina composite with ~10 vol. % SiC particulate additions. The hybrid NicalonTM/Alumina composite also exhibits a matrix microcracking yield strength of ~50 MPa, i.e., no apparent increase even though the microstructure exhibits a significant reduction in matrix microcracking. Furthermore, the hybrid NicalonTM/Alumina composite exhibits an ultimate tensile strength of ~190 MPa, i.e., a 35% reduction in the ultimate tensile strength compared to the standard NicalonTM/Alumina composite.

The lack of any significant increase in the matrix microcracking yield strength cannot be explained at this time. It was anticipated that the significant reduction in matrix microcracking would be accompanied by an increase in the matrix microcrack yield strength. However, a direct comparison of Figure 30 to Figure 31 is plotted in Figure 32 and shows that this did not occur. These data suggest that the ability of the composite to resist microcrack initiation and propagation under an applied load was not improved by the addition of the SiC particulates to the matrix, even though the occurrence of microcracks within the matrix was reduced. Further testing, characterization, and analysis is required to fully understand this phenomenon.

Figure 30. Stress-strain behavior of the standard Nicalon™/Alumina.

Figure 31. Stress-strain behavior of the hybrid Nicalon™/Alumina composite with 10 vol. % SiC particulate additions.

Figure 32. Comparison of stress-strain behavior of the Nicalon™/Alumina composites with and without SiC particulate additions.

The lower ultimate tensile strength of the hybrid Nicalon™/Alumina composite is understandable. The incorporation of SiC particulate into the Nicalon™ fiber preform resulted in a decrease in the volume fraction of Nicalon™ fiber in the resulting composite. Specifically, the standard Nicalon™/Alumina composite contains ~35 vol. % Nicalon™ fiber. The hybrid Nicalon™/Alumina composite with 10 vol. % SiC particulate contains only ~30 vol. % Nicalon™ fiber. The ultimate tensile strength of these composites is very dependent upon the volume fraction of continuous fiber in the composite. Therefore, the reduction in the ultimate tensile strength of the Nicalon™/Alumina composite after the addition of SiC particulate can be attributed to the reduction of the volume fraction of continuous Nicalon™ fiber within the resulting composite.

Tensile stress rupture tests were conducted on the hybrid NicalonTM/Alumina composites containing ~10 vol. % SiC particulates. The test results are summarized in Table 10. The stress level was periodically increased until failure. These data indicate that the maximum stress that the material can accommodate at 1100°C for extended periods of time is <100 MPa. A comparison of the stress-rupture results in Table 10 with the data presented in Figure 3 for the standard NicalonTM/Alumina composite indicates that the addition of SiC particulates had little effect on the tensile stress rupture behavior (oxidation characteristics) of the NicalonTM/Alumina composite.

Table 10. Tensile stress rupture of the NicalonTM/Alumina composites with 10 vol. % SiC particulate additions.

Test #	Temperature	Stress Level/Time
1	1100°C in air	69 h @ 80 MPa + 72 h @ 90 MPa + failure @ 100 MPa within 1 h
2	1100°C in air	67 h @ 80 MPa + 70 h @ 90 MPa + 67 h @ 95 MPa + 8 h @ 100 MPa - failure

In summary, microstructural evaluation of the hybrid NicalonTM/Alumina composite indicated that the SiC particulate additions significantly reduced the amount of microcracking in the matrix-rich regions of the composite. However, tensile testing indicated that the reduced matrix microcracking had little or no effect on the matrix microcrack yield strength. A definitive explanation for this observation cannot be made

at this time. The tensile testing also indicated that the hybrid NicalonTM/Alumina composite exhibited ~35% reduction in ultimate tensile strength compared to the standard NicalonTM/Alumina composite. This was attributed to the reduction in the volume fraction of NicalonTM fibers that resulted from the addition of the SiC particulates. The addition of the SiC particulates did result in a 20-25% increase in the through thickness thermal conductivity. This was attributed to the slight increase in the volume fraction of a higher thermal conductivity phase (SiC vs. Alumina) and the reduced matrix microcracking. The tensile stress rupture behavior of the hybrid NicalonTM/Alumina composite was very similar to the standard NicalonTM/Alumina composite and indicated that these materials can accommodate a maximum stress of <100 MPa at 1100°C for extended periods of time.

Task 5.3 HPZTM Fiber Reinforced Alumina (Task 3.1.2)

Initial preform optimization studies were conducted. Several 2-D woven HPZTM fiber preforms were given the same BN/SiC interface coatings that were developed for the NicalonTM/Alumina composite. Process feasibility was demonstrated on small 3 x 3" composite plates that were fabricated with 8 harness satin weave HPZTM fabric and the BN/SiC interface coatings. The microstructure of this new CFCC material system is shown in Figure 33. However, as the microstructure shown in Figure 33 indicates, microcracks propagated through instead of around the HPZTM fibers, which implies a strong fiber-matrix interface. At the time, it was suspected that the strong fiber-matrix interface was due to a possible reaction between the HPZTM fiber and the BN debond layer. A second concern was the oblong shape of the fiber, which allowed intimate fiber to fiber contact and subsequent canning or sealing of the fiber bundles during interface coating, as shown in Figure 34. Since these sealed areas were inaccessible to the matrix during directed metal oxidation, areas of porosity are observed within the fiber bundles.

Four point flexural tests were conducted on the HPZTM/Alumina composite. The average flexural strengths at 23 and 1200°C in air were 198 ± 6 MPa (3) and 173 ± 32 MPa (4), respectively. The flexural strength values were approximately 50% of the predicted or expected values, based on the reported strength of the HPZTM fiber.

Figure 33. Microstructure of HPZTM/Alumina composite showing microcracks propagating through the HPZTM fibers.

Figure 34. Microstructure of HPZ™/Alumina composite showing fiber alignment and contact resulting in porosity.

Room temperature fracture surfaces shown in Figure 35 exhibit fiber-matrix debonding in some areas, and strong fiber-matrix bonding in others. Fiber pull-out is observed and debonding, when it occurs, is observed at the BN-fiber interface. However, the strong fiber-matrix bond observed in some areas is in agreement with the microstructure presented in Figure 33 showing crack propagation through fibers. Strong fiber-matrix bonding results in premature fiber failure, since it promotes matrix crack propagation through, and not around, fibers. Hence, this lowers the overall composite strength.

The BN/SiC interface coating system used for the HPZTM/Alumina composite was the same as that used for the standard NicalonTM/Alumina composite. Although this system provided a weak interface and, hence, excellent properties for the NicalonTM/Alumina composite (flexural strength >400 MPa at 23 and 1200°C in air), the same was not observed for the HPZTM/Alumina composite (flexural strength <200 MPa at 23 and 1200°C in air). The absence of a weak interface has been attributed to the surface characteristics of the HPZTM fibers, which are predominantly silica rich and susceptible to reactions with the BN debond layer. Any reaction between the silica rich surface and the BN debond layer would lead to a strong fiber-matrix bond and poor composite strength.

A SiC chemical barrier layer was provided via CVI in an attempt to eliminate the possibility of any interaction between BN and the HPZTM fiber. The HPZTM fibers were first coated with SiC and then coated with the standard BN/SiC interface. The resulting interface coating of SiC/BN/SiC separates the BN from the fiber, as shown in Figure 36. However, microcrack propagation through the fibers was not eliminated. Evidence of crack propagation through the fibers was still observed although there also exist some regions of crack arrest at the fiber-matrix interface.

(a)

(b)

Figure 35. Fracture surface of HPZTM/Alumina composite tested in four point flexure at 23°C.

Figure 36. Microstructures of HPZ™/Alumina composite using a SiC/BN/SiC interface system.

Two additional processing techniques aimed at minimizing the HPZ™-BN reaction were evaluated. First, very thick coatings of the BN debond layer were applied to the HPZ™ fibers. The idea was that if excess BN was applied to the HPZ™ fibers, then a weak interface would be maintained even after some reaction with the fiber. Unfortunately, this technique was not successful, and cracks still propagated through rather than around the HPZ™ fibers. A second technique evaluated involved chemically treating the HPZ™ fibers in an HF acid etch prior to the CVI application of the BN debond layer. The purpose of the acid etch was to remove the silica rich phase from the surface of the HPZ™ fibers and hopefully reduce their reactivity. HPZ™ fibers were examined after exposure to hydrofluoric acid for varying periods of time. The scanning electron microscope (SEM) was utilized to evaluate the extent of silica removal

from the fiber and any possible fiber degradation during the HF treatment as a function of time. The results of this investigation indicated that the acid etching was only partially effective at removing the silica rich layer from the surface of the HPZ™ fibers. Some fiber-interface coating interaction was still observed and a weak interface was not maintained throughout the composite.

In summary, a strong fiber-matrix interface was observed in the HPZ™/Alumina composite and has been attributed to the surface characteristics of the HPZ™ fibers, which are silica rich and prone to reactions with the BN debond layer. Elimination of this HPZ™-BN reaction is a significant technical hurdle to the successful utilization of these fibers in a CFCC material system that relies on a BN debond layer for a weak fiber-matrix interface. If this reaction cannot be minimized or eliminated, and very little success has been achieved to date, then these fibers will have limited utility in this CFCC material system.

5.3 Almax™ Fiber Reinforced Alumina (Task 3.1.4)

Initial preform optimization studies were conducted. Various fiber sizings and preform binders were evaluated for their compatibility with the chemical vapor infiltration process. The same BN/SiC interface coatings that were developed for the Nicalon™/Alumina composite were applied to the Almax™ fiber preforms. Process feasibility was first demonstrated on 2-D, 0°/90°, 8 HSW Almax™ fiber preforms. Initial microstructures looked very encouraging. Figure 37 shows a representative microstructure of this new type of CFCC. Process optimization and characterization of the resulting composites was then conducted.

The AlmaxTM high purity alumina fiber is a 10 μm diameter fiber with extremely close packing within fiber bundles. A schematic illustration of the first AlmaxTM/Alumina composite developed on this program is shown in Figure 38. A significant fraction of the fibers within the bundle are in contact with each other. This resulted in bundle canning or sealing during the interface coating process. Hence, the interior of the bundles were inaccessible during subsequent matrix growth, which resulted in porosity (see Figure 37). This porosity leads to inefficient load transfer from the matrix to the fibers and hence, lower composite strength. A schematic of the desired microstructure is shown in Figure 39. Here, the majority of the fibers are individually surrounded by the matrix, thus promoting load transfer from the matrix to a greater percentage of fibers in the composite.

Figure 37. Microstructure of 2-D woven AlmaxTM/Alumina composite.

Figure 38. Schematic representation of Almax™/Alumina composite showing close packing of fiber bundles and porosity.

Figure 39. Schematic representation of Almax™/Alumina composite showing optimized microstructure with good densification.

The microstructure of the composites resulting from the initial fabrication attempts with the 2-D woven Almax™ fibers exhibited a significant amount of fiber contact and porosity within fiber bundles as shown in Figure 40 (similar to the schematic in Figure 38). Modifications and improvements in processing resulted in a more desirable microstructure as shown in Figure 41, with matrix growth within fiber bundles and between fabric plies. One of the changes made to reduce porosity within the fiber bundles was a reduction of the number of filaments per bundle from 1000 to 500. Other processing improvements will not be documented here due to their proprietary nature.

Tests were conducted to evaluate the effectiveness of the BN/SiC interface coatings in the Almax™/Alumina composite. Microcracks were introduced in the composite by means of a microhardness indenter in order to study their interaction with the interface. A microhardness indentation using a 5 kg load is shown in Figure 42. The microcracks generated in the matrix propagate into the Almax™ fiber bundles. A higher magnification (Figure 42b) shows the effectiveness of the BN/SiC interface in deflecting the cracks and inhibiting crack propagation through the fibers. Scanning electron microscopy conducted on the same region (Figure 43) shows crack deflection at the BN-fiber interface, similar to what is commonly observed in the Nicalon™/Alumina composite.

Figure 40. Microstructure of the Almax™/Alumina composite showing canning and porosity within fiber bundles.

Figure 41. Microstructure of the optimized Almax™/Alumina composite showing less canning, reduced porosity and improved matrix densification.

(a)

(b)

Figure 42. Microstructure of the AlmaxTM/Alumina composite showing microcracks introduced via a microhardness indenter and the interaction of the microcracks with the interface coating.

Figure 43. Scanning electron micrograph showing crack deflection at the BN/AlmaxTM interface.

Average four point flexural strengths were measured from 3-3x3" flat plates and are summarized in Table 11. The average room temperature flexural strength of ~250 MPa was expected, considering that the NicalonTM/Alumina composite flexural strength averages a little over 400 MPa and that the strength of AlmaxTM fiber (1.8 GPa) is a little over half the strength of NicalonTM (3 GPa). Representative load-deflection curves from four-point flexural tests are shown in Figures 44 through 47. The deviation from linearity, observed in all cases during loading of the specimen, signifies matrix microcracking and fiber-matrix debonding. The tail observed in some of the high temperature load-deflection curves suggests that the weak fiber-matrix interface observed at room temperature is maintained at elevated temperatures.

Table 11. Flexural strength of the Almax™/Alumina composite.

Plate #	Fiber Vf	Average Strength (MPa) ± Std. Dev. (# of samples) at:			
		23°C	700°C	900°C	1000°C
1	—	259 ± 38 (4)	—	—	100 ± 26 (4)
2	0.40	248 ± 6 (4)	186 ± 13 (4)	150 ± 14 (4)	133 ± 32 (4)
3	0.37	246 ± 32 (5)	151 ± 5 (4)	—	104 ± 15 (3)

Figure 44. Load-deflection curves from flexural tests on the Almax™/Alumina composites at 23°C in air.

Figure 45. Load-deflection curves from flexural tests on the Almax™/Alumina composites at 700°C in air.

Figure 46. Load-deflection curves from flexural tests on the AlmaxTM/Alumina composites at 900°C in air.

Figure 47. Load-deflection curves from flexural tests on the AlmaxTM/Alumina composites at 1000°C in air.

The four point flexural strengths measured for the AlmaxTM/Alumina composite averaged approximately 250 MPa at 20°C, 170 MPa at 700°C, 150 MPa at 900°C and 110 MPa at 1000°C. Fracture surface analysis was conducted using the SEM in order to explain the significant drop in composite strength with increasing test temperature.

Composite fracture surfaces are shown in Figures 48 through 52 as a function of test temperature. Fiber-matrix debonding and fiber pull-out are observed in 0° as well as 90° oriented fibers for all samples irrespective of test temperature. This confirms that a weak fiber-matrix interface is maintained from 23 to 1000°C. The relatively small degree of fiber pull-out in the 0° oriented fibers is attributed predominately to the rough surface of the AlmaxTM fiber (Figure 52) which can lead to some degree of mechanical interlocking.

(a)

(b)

Figure 48. Fracture surface of AlmaxTM/Alumina composite after flexure testing at 23°C showing debonding and fiber pull-out (a=0°, b=90° fibers)

(a)

(b)

Figure 49. Fracture surface of AlmaxTM/Alumina composite after flexure testing at 700°C showing debonding and fiber pull-out (a=0°, b=90° fibers)

Figure 50. Fracture surface of Almax™/Alumina composite after flexure testing at 900°C showing debonding and fiber pull-out.

(a)

(b)

Figure 51. Fracture surface of AlmaxTM/Alumina composite after flexure testing at 1000°C showing debonding and fiber pull-out (a=0°, b=90° fibers)

Figure 52. Fracture surface of Almax™/Alumina composite after flexure testing at 700°C showing rough fiber surface.

An evaluation of fiber fracture surfaces (Figures 53 through 55) show a typical transgranular type of failure at room temperature (Figure 53). The fine porosity observed explains the relatively low density of the Almax™ fiber (3.6 g/cm³) versus that of pure alumina (3.97 g/cm³). The porosity would also account for the relatively low strengths observed at room temperature. However, a fiber fracture surface from a sample tested at 1000°C (Figure 54) exhibits a completely intergranular type of failure. This suggests weak grain boundaries at these temperatures and implies the presence of weak or low melting grain boundary phases. The change in fiber fracture morphology from transgranular to intergranular between 23 and 1000°C accounts for the 50% drop

in composite strength observed between the two temperatures. The decreased amount of porosity observed in the fiber fracture surfaces at 1000°C versus those at room temperature suggests the presence of pores within the grains and not at the grain boundaries. Fiber fracture surfaces from samples tested at 700 and 900°C (Figure 55) exhibit a mixed mode of failure. The degree of intergranular type failure at these intermediate temperatures could not be quantified.

(a)

(b)

Figure 53. Fracture surface of AlmaxTM fiber after flexure testing at 23°C exhibiting transgranular failure mode and porosity.

(a)

(b)

Figure 54. Fracture surface of AlmaxTM fiber after flexure testing at 1000°C exhibiting an intergranular failure mode.

(a)

(b)

Figure 55. Fracture surface of AlmaxTM fiber after flexure testing at (a) 700°C and (b) 900°C exhibiting a transition between transgranular and intergranular failure.

The Almax™ fiber grain size is between 0.5 and 1.0 μm . Hence, creep and grain growth are also issues that needed to be addressed for high temperature applications. Composite samples were exposed to 1000°C for 100 h in air in order to evaluate fiber grain growth and strength retention. The results, summarized in Table 12, show a small drop in strength at room temperature as a result of the heat-treatment. The 1000°C strength was unaffected.

Table 12. Flexural strength of the Almax™/Alumina composite before and after heat treatment.

	Average Strength (MPa) $\pm$ Std. Dev.		
	(# of samples) at:		
	20°C	700°C	1000°C
As-fabricated	259 $\pm$ 38 (4)	—	100 $\pm$ 26 (4)
Heat-treated	209 $\pm$ 18 (3)	139 $\pm$ 16 (3)	108 $\pm$ 13 (3)

Two additional 6x6" flat plates were fabricated and machined into test coupons for continued mechanical property evaluation. The data are presented below in Table 13.

Table 13. Material properties of the AlmaxTM/Alumina composite.

Property	Test Temperatures		
	23°C	700°C	1000°C
Flexural Strength (MPa)	251 ± 27 (8)	186 ± 13 (4)	117 ± 29 (8)
Tensile Strength (MPa)	130 ± 12 (5)	—	—
Modulus (GPa)	155 ± 10	—	—
Failure Strain (%)	0.11 ± 0.02	—	—
Proportional Limit (MPa)	50 ± 13	—	—
Fracture Toughness (MPa√m)	19.2 ± 2 (5)	6.6 ± 1 (5)	3.6 ± .3 (5)

In summary, the mechanical property testing conducted on the AlmaxTM/Alumina composite indicated that both the strength and toughness of this composite dropped significantly with increasing test temperature. This was attributed to a degradation of the AlmaxTM fiber and the resulting change in fiber fracture mode from transgranular to intergranular. This oxide fiber-oxide matrix composite may offer good potential for increased oxidation and corrosion resistance over the NicalonTM/Alumina composite due to the more stable AlmaxTM oxide fiber. However, an oxide fiber with improved high temperature strength is required before the full potential of an oxide fiber-oxide matrix CFCC material system can be realized.

5.4 Chopped Nicalon™ Fiber Reinforced Alumina

Chopped Nicalon™ fiber mats were used to fabricate 3x3" plates. The BN/SiC interface coatings were used as a fiber-matrix interface. Initial studies were directed towards optimizing the volume fraction of the chopped fibers and varying the CVI process conditions to obtain the desired interface coating thickness.

A representative microstructure is shown in Figure 56 illustrating the characteristics of this new, innovative, low cost CFCC material system (and demonstrating process feasibility). This composite is characterized by a low volume fraction of chopped Nicalon™ fiber (~10 vol.%) due to the low volume fraction of chopped fiber in the mats used for the preform. This results in a relatively large volume fraction of matrix (~80 vol.%), as is evident in the low magnification microstructure in Figure 56a. It is important to note that there is very little evidence of matrix microcracking apparent in Figure 56. Additional 3x3" plates were fabricated and machined into flex bars for preliminary mechanical property determination. Larger 6x6" plates were fabricated once the CVI coating conditions and matrix growth parameters were optimized.

Process improvements on the 6x6" flat plates resulted in an increased fiber content to 15 vol.%. Representative optical micrographs (Figure 57) exhibit no significant matrix microcracking despite the large mismatch between fiber (~4 ppm/°C) and matrix (~8 ppm/°C). The reduced matrix microcracking is attributed to the random distribution of fibers in the matrix.

(a)

(b)

Figure 56. Microstructure of the chopped NicalonTM/Alumina composite with ~10 vol. % chopped fiber.

(a)

(b)

Figure 57. Microstructure of the chopped Nicalon™/Alumina composite with ~15 vol. % chopped fiber.

Flexural test specimens were machined and tested to evaluate composite strength at room and elevated temperatures. The data is presented below in Table 14.

Table 14. Flexural strength of the chopped Nicalon™/Alumina composite.

Test Temperature °C	Strength (MPa)	No. of Samples
23°C	114 ± 33	5
600°C	106 ± 22	5
1200°C	115 ± 14	5

These data indicate that this composite does not exhibit good room temperature or high temperature flexural strength. This can be attributed to the relatively low volume fraction of chopped Nicalon™ fibers in the composite. Therefore, if actual components were fabricated utilizing chopped Nicalon™ fiber for the reinforcement phase, an attempt would have to be made to increase the volume fraction of chopped fiber in the preform in order to provide adequate mechanical integrity in the composite. This would require the development of suitable preforming techniques capable of producing chopped Nicalon™ fiber preforms with higher volume fractions of chopped fiber.

6.0 DU PONT LANXIDE COMPOSITES - DIMOX™

- A new, safe, scaleable CVI process was utilized to deposit protective SiC interface coatings in the commercially available Nicalon™/Alumina composite. These composites exhibited improved interface coating uniformity and mechanical properties equal to or better than the target values. This Nicalon™/Alumina composite provides the strength, toughness, and environmental stability required for the intended end-use applications and will be scaled-up during Phase II of the DOE-CFCC Program.
- Process feasibility was demonstrated on a hybrid Nicalon™/Alumina composite that contained ~10 vol. % SiC particulate. The incorporation of SiC particulate into the Nicalon™/Alumina composite resulted in a 25% increase in the thru thickness thermal conductivity. In addition, microstructural evaluation indicated that the SiC particulate additions significantly reduced the amount of microcracking in the matrix-rich regions of the composite. However, the observed reduction in matrix microcracking did not appear to increase the matrix microcrack yield strength.
- Process feasibility was demonstrated on an HPZ™/Alumina composite that contained ~35 vol. % HPZ™ fibers in a 2-D, 0/90°, 8 HSW architecture. However, this composite exhibited low flexural strengths due to the strong bond between the BN interface coating and the HPZ™ fibers. This strong interface is attributed to reactions between the oxygen rich fiber surface and the BN

coating. Attempts to minimize or eliminate this interaction have not been successful to date.

- Process feasibility was demonstrated on an AlmaxTM/Alumina composite that contained ~35 vol. % AlmaxTM fibers in a 2-D, 0/90°, 8 HSW architecture. This composite exhibited good room temperature strength and toughness, but reduced properties with increasing test temperature. The low composite strength and toughness at elevated temperatures is attributed to a decrease in fiber strength. Fiber fracture surfaces exhibited an intergranular mode of failure at high temperatures, which suggests the presence of low melting point grain boundary phases.
- Process feasibility was demonstrated on a chopped NicalonTM/Alumina composite that contained 10-15 vol. % of the chopped fiber. As expected, the low fiber volume fraction resulted in low composite strengths. However, the room temperature flexural strengths were retained at 1200°C in air.

7.0 WESTINGHOUSE HOT GAS FILTRATION SYSTEMS

Task 1 Application Assessment and Benefits

Hot gas cleaning systems provide a low risk opportunity to enhance development of a U.S. continuous fiber reinforced ceramic composite (CFCC) manufacturing base. Development of a porous CFCC filter element will beneficially support national programs, by improving control of industrial pollutants and by improving the economics of generation of power using advanced fossil fuel power plants.

The definition of hot gas cleaning systems or hot gas filtration, in the context of this work, is using a barrier filter for very high efficiency (>99.9%) particulate removal, in the temperature range 300°C (575°F) up to basically 1000°C (1850°F). This, compared to high temperature filtration of gaseous streams using various cloth media such as glass, Teflon®, Ryton®, etc. Additionally, hot gas cleanup embraces concepts beyond particulate removal, e.g., the removal of chemical contaminants using solid sorbents, introduced upstream of the barrier filter, which react with the contaminant and are then removed by the filter.

The benefit of a CFCC hot gas filter for the industrial sector is its potential robustness and chemical inertness over a wide range of conditions that are representative of industrial process gas streams. A strategic feature of the Westinghouse/DuPont commercialization plan is to leverage Westinghouse's current participation in the development of hot gas filter (HGF) systems for advanced fossil based utility power generation, to provide CFCC filter testing and qualification. This

will accelerate acceptance by the industrial sector and reduce risk and development cost.

A highly efficient, robust, long-lived filter system capable of operating at temperatures of about 1800°F is an essential element of highly efficient combined cycle plants burning coal. The filter system is necessary not only to reduce pollutants, but also to lower the risk of eroding and corroding vital turbine parts. The system must operate at as high a temperature as practical otherwise plant efficiency is seriously impacted. Westinghouse is a key player in this critical technology by providing HGF systems and turbines, as well as combustors.

Westinghouse is an integrated system supplier of hot gas filter technology to the fossil fuel-gas turbine power plant. Currently we are expanding the utilization of this technology into metals processing, petrochemicals and the waste-to-energy sectors. Figure 58 shows the Westinghouse system for a ceramic barrier filter vessel used in pressurized hot gas filtration. The refractory lined filter vessel contains the filter elements (in this case, candles) mounted in modular cluster assembly. This modular approach facilitates maintenance and system scaleup. The cluster assemblies are hung from a load bearing, hot metal structure which consists of a tubesheet and expansion cone assembly.

Currently we use monolithic porous ceramic candle or cross-flow filter elements. These are limited in their ability to handle large thermal transients and dynamic mechanical loads that can occur during process upsets. Thus, a strong, more damage tolerant and environmentally stable filter element would be of decided advantage in commercial application. The ability of current filters to handle a large number of blow-

back cleaning cycles may also be limited due to the stress created by thermal transients. Additionally, to minimize corrosion and heat loss, Westinghouse uses an uncooled tubesheet which limits process conditions to less than 1650°F due to loads on the hot metal structures. In some advanced coal gasification and industrial processes, it is advantageous to have operating temperatures above 1650°F. The availability of high temperature alloys for this kind of service is very limited. CFCC's would be beneficial in both of these areas—candles and hot load bearing structures.

Figure 58. Hot gas cleaning systems - Westinghouse candle filter system.

Recognizing the improvements in material properties provided by CFCCs compared to monolithic ceramics, one can conceptualize the advantages that CFCCs may bring to the HGF system supplier. This is outlined in Table 15.

Table 15. HGF system benefits derived from the use of CFCCs.

System Driver	CFCC Attributes
Design Flexibility <ul style="list-style-type: none">• Thermal Transients• Array/Attachment Design• Wider Operating Envelope (Temp./Gas Composition)	<ul style="list-style-type: none">• Thermal Shock Resistance• Strength• Creep Resistance• Inert Material
Increased Capacity <ul style="list-style-type: none">• Lower Pressure Drop• Longer Length	<ul style="list-style-type: none">• Thin Wall• Strength
Longer Life	<ul style="list-style-type: none">• Thermal Shock Resistance• Strength• Inert Material

The market potential, which represents the size of the market, which from a technical need, stands a reasonable chance of being a target for CFCC filter media is given below. Clearly, the actual penetration by CFCC or any other filter media will depend entirely on availability, successful demonstrations, data on long-term durability, and pricing.

The data below is summarized (ref. Table 15.a.) from the prior discussions and represents either current market size of near-term (1998) market development.

Table 15.a. Filter media market size - 1998.

	Filter Media Required		
	Filtration Area (ft. 2)	\$ Sales	
		Current	
		Rigid Ceramic Filter Price (\$ M)	\$200/Sq. Ft. Price (\$ M)
Advanced Fossil Fuel Applications	50,000	9.7	12
Waste Incineration	120,000	19	25
Fluid Cat. Cracker Regenerators	750,000	120	150
Chlorinators in Extractive Metallurgy	90,000	14.5	18
Reduction to Volatile Metals	<u>250,000</u>	<u>41</u>	<u>50</u>
TOTAL	1,260,000	204.2	255

If 20% of this target can be realized by the year 1998, the potential size of CFCC production will have to be about 114,000 sq. ft., which at current sales prices is about \$40M which is reasonably close to the \$35M figure estimate obtained from Reference 1 for the high performance end of the filter media market for HGF.

Task 1.2 Application Definition and Performance Requirements

In this task we define the performance requirements for CFCC filter elements to be used in HGF systems. In Task 1 we identified several different market segments in which ceramic and, ultimately, CFCC filters would have the potential to penetrate. With each of those opportunities, a set of criteria was presented which define the

operating envelope for the filter medium. This of course covers a wide range of temperatures, pressures and gas and particulate species. To define the performance requirement for CFCC filters, we have chosen the operating conditions for advanced fossil fuel applications because the conditions are stringent and we have the most experience with the. Westinghouse commercialization strategy for CFCC filters is to leverage Westinghouse's participation in the development of HGF systems for advanced fossil-based utility power generation to provide an opportunity for CFCC filter testing and qualification.

Materials Evaluation

Samples of 1/8" thick laminates made by the DuPont-Lanxide Dimox™ process consisting of 2D Nicalon™ fiber/Alumina matrix were subjected to porosity tests and were found to have no detectable flow of gas through them even when subjected to a ΔP of 14.5 psi. Since this material could not be made porous enough to serve as a filter, it was dropped from further evaluation and attention was devoted to the DuPont Lanxide CVI SiC/SiC CFCC candidate material.

Samples of a prototype element of DLC CVI SiC/SiC were subjected to C-ring compression and tension tests and the results, which were found to be substantially higher than for current monolithic filters (1,500-3,500 psi).

The prototype CVI SiC/SiC candle filter element from DuPont Lanxide Composites was also subjected to testing as a filter element in our high temperature/high pressure loop. The initial prototype performed well as a filter with <1 ppm of dust in the outlet—well within the nominal specifications (ref. Table 15.b) for our HGF requirements. The pressure drops and flows were well within our specification (ref. Table 15.c).

Because the initial results are very encouraging, further development of this filter will be conducted in Phase II of this program.

Table 15.b. Candle filter technical specifications.

Item	
Length	1515 $\pm$ 10mm
Linearity of Filter Body	Bowing not to exceed 3mm end-to-end
Concentricity	$\pm$ 2mm
Perpendicularity of Flange to Body	$\pm$ 0.5°
Weight	To be provided by vendor
Strength	
C-Ring Compression	
4-Point Bend (RT)	To be provided by vendor
4-Point Bend (870°C)	
Initial Pressure Drop	6 $\pm$ 2 mbar at 52 m ³ /hr/candle (70°F, air)
Particle Collecting Efficiency	>99.9%
Bursting Pressure	>60 bar
Ultrasonic Time-of-Flight at 200 kHz (end-to-end)	<190 μ s/m
Patch or Filter Repairs	None allowed

Table 15.c. Operating conditions and filtration requirements.

	Combustor	Gasifier
Temperature (°F)	1650	1800
Pressure (psia)	150-300	150-320
Composition	Oxidizing CO ₂ , O ₂ , N ₂ Trace: SO _x , NO _x , alkali metals	Reducing Co, H ₂ , N ₂ Trace: NH ₃ , HCN, H ₂ S alkali metals
Particulate Loading (ppmw)	1,000-20,000	2,000-40,000
Max. Outlet Loading	<20	<20
HGF ΔP (psia)	<7.5	<10
HGF ΔT (°F)	<10	<10
Filter Durability	3 Yrs, 100 Startups	3 Yrs, 100 Startups

Task 1.3

Design Impact

In this task we define the design impact on Hot Gas Filter (HGF) systems from the availability of CFCC materials. Two areas were investigated: 1) the filter element, and 2) the tubesheet which, in the Westinghouse design, is hot and supports the clusters and filters and also seals the clean side of the filter vessel from the hot side.

Filter Element

It can be assumed that a filter element made from a porous CFCC material when compared to its monolithic counterpart would be stronger, stiffer and have higher fracture toughness. The use of a CFCC filter element would have as a major impact

increased durability in use, particularly as it relates to the ability to absorb thermal transients caused by backpulsing with cold gas during cleaning and due to process upsets, without undergoing micro- and macro-cracking. Secondary impact would come about from reduced weight of a stronger filter material which would reduce the mechanical requirements for the plenum and/or allow the use of much longer individual candles which in turn would permit a more efficient and more economical filter vessel design.

A real assessment of the durability of a CFCC filter element must await the detailed testing that will be accomplished in the Phase II Program. In order to gauge the durability of the DuPont CVI SiC/SiC material, we used the material properties given to us by DuPont, in an energy balance model which we have successfully used in the past to analyze transient stresses in a candle during pulse cleaning, and during process upsets. The model generates temperature profiles as a function of time and distance from the filter wall surface, and from that profile calculates stress in the filter element, as a function of time and distance from the wall. The air temperature upset from steady state is considered to be caused by back pulsing with cold air or N_2 , or by process transients. For input to the model we used actual data on thermal transients obtained from the Westinghouse tests conducted in pilot facilities.

It is seen that the resulting maximum strain at any point and time is well below the strain-to-failure (0.4%). The maximum point strain is about 0.13% at the candle ID wall, for the pulse gas transient case.

The analysis indicates that the DuPont CVI SiC/SiC material at least has the right mix of thermal and mechanical properties to be a good starting point for a durable candle filter element. Additionally, we can observe that the CFCC material has twice

the fracture toughness of the current monolithic filter which would indicate a higher level of durability.

A more durable filter element, which does not require frequent replacement, can have a significant impact on the operating costs of an advanced fossil fuel power plant. This can be estimated as follows.

A commercial scale 250 MWe integrated gasifier combined cycle (IGCC) plant would have a gas flow of 28,000 acfm and would require about 5,740 sq. ft. of filter surface or about 1980 candles of current size and geometry. If the CFCC candle costs 50% premium (i.e. \$225) over today's monolithic candle then the additional material cost would be \$450K. However, the replacement requirements would be reduced. If we assume that the current ceramic candle charge needs to be replaced when 15% of the candles break ("Failsafe" designs would block the exit from the broken candle and therefore the gas flow through undamaged candles would increase up to 15%) then we can estimate the cost of each replacement event, for current ceramic candles.

Cost of candle @ \$450/unit =	\$134K
Labor, 168 hrs x 3 laborers x \$35/hr =	\$18K
Replacement power @ 2 cents/k Wh	
168 hrs x 250,000 kW x 0.02 =	<u>\$840K</u>
Total cost for the event	\$992K

Thus it can be seen that if the use of the more robust and durable CFCC candles were to reduce even one replacement event/year the savings would be significant even if the premium for the CFCC candle were to double.

CFCC Tube Sheet

The material used in this application would be a dense CFCC and the alumina matrix CFCC produced by the DuPont Lanxide DIMOX™ process would be the material of choice particularly for the oxidizing atmosphere of combustors.

The metal tubesheet design used by Westinghouse utilizes hot metal structures made from high nickel alloys costing around \$8-10/lb or \$3/cu.in. for 1600°F service. However, for filter service at 1800-1900°F which could be required for high efficiency fossil fuel plants in the near future one has to specify very exotic alloys whose price may run up to \$30/lb. (\$10/cu. in.) and whose availability in required sizes may be limited. Therefore, if it is possible to mold a tubesheet made from CFCC in the sizes required (6-16 ft. in diameter), then their availability could prove advantageous for HGF applications at temperatures of >1650°F.

Any design for a hot tubesheet, which is supported at its circumference, by being clamped between the flanges of a cold vessel, needs to contend with the thermal stresses created by the differential thermal expansion. In the standard Westinghouse hot metal tubesheet design, the differential thermal expansions are absorbed by means of an expansion cone which absorbs the strain by bending and in creep relaxation. To determine the feasibility of using a CFCC tubesheet, it was necessary to first determine how the CFCC tubesheet would be coupled to the cold walls of the filter vessel.

Several different configurations of a 10 ft. diameter CFCC tube sheet with a 3 cluster deadweight load were analyzed using the WECAN/Plus finite element program. A coupled thermal analysis and static analysis were performed on the seven different models to determine the thermal stresses in the tubesheet. The goal of this effort was

to arrive at a configuration which would limit the maximum stress to <12 ksi, the stress rupture for the 2D NicalonTM/Alumina CFCC.

For the thermal analysis the top and bottom surfaces of the tube sheet were constrained to 1800°F from 0 to 51 inch radius. The end of the tubesheet (at 60 inches) was constrained to 100°F. The resulting temperature distribution was solved and read for a stress analysis run. For the static analysis the end of the tubesheet (at 60 inches) was coupled to move in the horizontal plan as a unit and the center point was constrained to not move in the vertical direction.

Since resulting stresses were concentrated at the large thermal gradient between 51 inch radius and the 60 inch radius, various configurations between these radii were analyzed to reduce stresses due to the high thermal gradients.

It is possible to design a working tubesheet using a CFCC material which would reduce the tubesheet weight to 10% of an equivalent metal tubesheet. However, since no simple configuration of end joint works, it is difficult to determine the economic viability of using a CFCC material for the tubesheet since cost of processing complex shapes are hard to determine.

Task 1.4

Commercialization Plan

The Westinghouse commercialization plan for CFCCs involves integration with our HGF systems to create a ready market sector for CFCC filter elements. As a product line, these elements can serve large user segments within and outside the HGF sector.

Development and commercialization of CFCCs for Westinghouse hot gas filter systems enhances U.S. competitiveness through production of high-efficiency, durable long-life filter elements which, when implemented, provide process advantages and particle removal efficiencies to meet Clean Air Act requirements.

High-performance ceramic barrier filters are projected to play a significant role on the near-term and future growth of the U.S. utility industry. Utility investments in HGF systems will be enhanced by the projected improvement in reliability using CFCC filter elements. This improvement is highly leveraged by plant availability and couples well with the market-driven advantage of hot gas filtering, which is essential for advanced fossil fuel cycles such as Integrated Gasifier Combined Cycles, in which filters protect downstream turbines and other equipment. In these applications, hot gas filter systems achieve Clean Air Act standards but with savings through the improved efficiency of combined cycles. In addressing this market, reliability improvement alone will drive replacement of filter elements for improved plant availability. Hence, an ideal market-driven commercialization opportunity exists for CFCC filter elements in the parallel Westinghouse commercialization of HGF systems.

Table 16. Outline of Westinghouse commercialization plan for CFCC hot gas filters.

<u>Commercialization Program Elements</u>	<u>Westinghouse Approach for Selected Application</u>	<u>Benefits of Approach</u>
<ul style="list-style-type: none"> • Technology Development 	<ul style="list-style-type: none"> • Focused and application driven • Utilize W experience from fossil energy development activities to support total system development. 	<ul style="list-style-type: none"> • Minimum development cost and time; W as OEM drives product development, captures spin-offs.
<ul style="list-style-type: none"> • Product Qualification 	<ul style="list-style-type: none"> • CFCC filter elements are implemented into ongoing test and development programs. 	<ul style="list-style-type: none"> • Qualification path defined; increases likelihood of early industry acceptance.
<ul style="list-style-type: none"> • Targeted Markets 	<ul style="list-style-type: none"> • Utility and Industrial Sector 	<ul style="list-style-type: none"> • Synergism of markets captured. True need exists based on limitation of conventional technology or identification of significant process/environmental advantage. • First cost issued minimized.
<ul style="list-style-type: none"> • Manufacturing/Production Facilities 	<ul style="list-style-type: none"> • Utilize DuPont existing facilities • Production capabilities expanded to meet product/market growth. 	<ul style="list-style-type: none"> • Immediate capitalization needs are minimized.
<ul style="list-style-type: none"> • Marketing/Sales 	<ul style="list-style-type: none"> • Implemented through existing W/DuPont organizations. 	<ul style="list-style-type: none"> • Established customer relationships; also provides lowest dollar impact organization growth.

Marketing Plan

The basic application proposed herein relies on the simplest possible marketing plan; substitution in Westinghouse products of a superior CFCC component, and making this component universally available. The potential for order-of-magnitude reliability improvements suggests that this approach will be very effective.

Within Westinghouse, environmental opportunities are now recognized at the Group level. The Environmental Systems Group has been organized at the same levels

as the Energy and Power Generation Group. This emphasis yields a second-phase implementation following utility application within Westinghouse, minimizing start-up marketing investments since the Science & Technology Center is also networked into the Environmental Systems Group strategic program.

8.0 SOLAR TURBINES FOR INDUSTRIAL GAS TURBINES

Task 1 Application Assessment

The objective of the program is to provide DuPont Lanxide Composites Incorporated with an assessment of the use of their NicalonTM reinforced alumina matrix composite in selected gas turbine applications. This feedback is intended to enhance and expedite DuPont Lanxide's ability to develop and commercialize their continuous fiber reinforced ceramic matrix composites.

The Phase I Solar subcontract was divided into three tasks. The first task involved the analysis of selected applications in terms of performance, energy savings, economic benefits, strategic factors, and environmental effects. The turbine applications that initially were to be studied included both natural gas fired components (combustor and nozzle) and coal fired turbine system components (Particle rejection impact separator, candle filters, and transfer tubing). Since coal-fired applications do not reflect the current goals for Solar's product line, the program scope was redefined to focus on only the gas turbine applications.

The Task 2 addressed the design definition of the nozzle and combustor applications. Task 3 includes the analysis of conceptual design layouts for the combustor and the nozzle.

Task 2.0

Combustors For Industrial Gas Turbines

The combustion system, a key component of the gas turbine engine, produces thermal energy from the chemical energy of fuel at high temperatures and pressures. To generate this thermal energy the combustor utilizes compressor supplied air in three different ways. Firstly, reaction air is required to oxidize the fuel. Secondly, dilution air is required to both reduce the overall temperature of the gases entering the turbine section and to tailor the temperature distribution of those gases for optimum turbine design. Thirdly, a significant amount of air is utilized to film-cool the combustor liner, refer to Figure 59.

With conventional sheet metal alloys up to 50% of the compressor supplied air is directed toward the combustor side walls in order to maintain structural integrity and ensure component life. A significant increase in turbine inlet temperature is incompatible with the film cooling process using standard wall materials. Materials with improved durability (compared to metals) could advantageously be used in the combustion liner to reduce the need for increased film cooling air at elevated turbine firing temperatures.

These factors point to several benefits of the application of oxide based CFCCs in combustors:

- a. Oxide CFCC materials can replace metals in current combustor liners that are limited in service life because of oxidation damage.
- b. CFCC materials will reduce or eliminate the need for film cooling air and increase the amount of the dilution air which will reduce emissions.

- c. The increased availability of dilution air is expected to improve the pattern factor of the combustor. This will lessen the occurrence of hot spots in the first stage nozzle area of the gas producer turbine, improve nozzle durability and/or allow increased firing temperatures.

Figure 59. Schematic of a typical combustor.

To fully eliminate the film cooling requirement, the wall material must operate in an oxidizing environment with temperatures of 1371°C (2500°F) and above, a substantial increase compared to the 871°C (1600°F) maximum temperature of current metallic combustor liners in typical industrial engines. The material requirements are the ability to fabricate the complex geometries needed for an efficient energy conversion device and thermo-mechanical compatibility with the adjoining engine components.

Gas Turbine Nozzles

There are opportunities for applications of oxide based CFCCs in the hot section of gas turbines. Uncooled, oxide based CFCCs could be used in gas turbines fired with conventional fuels to replace air-cooled metallic nozzles that operate at metal temperatures of 1600°F or below. Another field of application will be gas turbines operating with lower grade fuels containing aggressive contaminants. Fabricating these components from oxide based CFCCs would be a novel approach to service life enhancement in the turbine of multi-fuel combustion systems.

The use of ceramic matrix composites as structural materials for the turbine hot section for engines using a non-conventional fuel (e.g., coal) has been the subject of a recent DOE sponsored study. The study concluded that airfoil shaped nozzles vanes, ref. to Figure 60, and rotor blades in the turbine hot end offer the greatest potential in terms of potential increases in system performance. Greater thermal efficiencies and output power can be obtained than are currently achievable with metallic components.

Figure 60. Conceptual design of ceramic turbine nozzle vane.

Task 3.0

Impact Assessment

The applications selected for analysis include combustor liners and nozzles. This work is designed to consider the potential impacts resulting from introduction of components made of DuPont Lanxide CFCC and to help guide the development of the material by assessing the design needs of components. The areas of potential impacts will include performance, energy savings, economic benefits, strategic factors, and environmental effects. These impact areas represent critical factors governing the marketing of CFCCs.

Gas Turbine Engine Development

Three generations of engine design approaches have been considered for the introduction of ceramics into industrial gas turbines. These generations are:

First Generation Engines

Replacement of the cylindrical portions of the metallic annular combustor by ceramics, plus replacement of ceramic nozzles and ceramic blades in the Solar Centaur 50 engine is an example of a first generation engine. In order to achieve the desired increase in efficiency on increasing the firing temperature of the Centaur 50 model from a total rotor inlet temperature (TRIT) of 1850 to 2050°F. The expected increase in efficiency was realized.

Second Generation Engines

The second generation of engines to be introduced into the marketplace will follow the first generation by an interval that will depend on the progress of the first generation. The firing temperatures will be higher (e.g., TRITs of 2200°F) but the number of stages to be converted to ceramics will be similar to that of the first generation.

Third Generation Engines

Complete optimization of the gas turbines will be performed at this time to take full advantage of ceramic components including CFCCs. Higher temperatures and more ceramic components would be expected to be introduced.

Gas Turbine Engine Performance

All engine performances relate to standard (ISO) conditions without duct losses, without water injection to control NO_x, for sea level with 59°F air at 60% R.H. (in ISO conditions).

Table 17 compares the average performance of the metallic Centaur 50 with that predicted for the ceramic CSGT Centaur 50 engine under IISO conditions.

The ceramic modification of the Centaur 50 gives 25.9% more power and 5.6% increase in efficiency. In the cogeneration mode, the electrical generation efficiency increases by 5.3% and the fuel utilization efficiency increases by 6.6%. The electrical output increases by only 25.6% because of the back pressure created by the heat recovery steam generator (HRSG).

Table 17 Comparison of metallic centaur 50 fired at 1850°F and ceramic CSGT centaur 50 fired at 2050°F.

Performance Factor	Average Metallic Centaur 50	Predicted Modified Ceramic Centaur 50 (SCGT)	Increase (%)
<u>In Simple Cycle Mode:</u>			
Total Rotor Inlet Temp. °F	1850	2050	—
Shaft Output, SHP	5880	7402	25.9
Shaft Thermal Eff. %	29.6	31.3	5.6
Electrical Output, KW	4144	5217	25.9
Electrical Thermal Eff. %	28.0	29.6	5.6
<u>In Cogeneration Mode (No Supplementary Firing):</u>			
Electrical Output, KW	4051	5088	25.6
Electrical Thermal Eff., %	27.4	28.8	5.3
Steam Output, pph	20,300	25,950	27.8
Overall Thermal Eff., %	75.4	80.3	6.6
Electrical Thermal Ratio	0.57	0.56	0

Market Outlook

Product characteristics that customers perceive as valuable in the 1990's include reliability, availability, and maintainability (RAM), first cost, efficiency, and emissions. In the year 2000 and beyond, the prevalent trend will be an emphasis on reduced emissions and increased efficiency. First cost will not be as pertinent as in the 1990's.

Based on the energy supply and demand forecast, the key market variables and the buying criteria, the base case market outlook for industrial gas turbines is contained in Table 18. It does not include gas turbines for transportation applications

(marine, land or air). It assumes evolutionary gas turbine development throughout the planning period (2010). Major technical breakthrough which may result from programs are not expected to make a significant change to the outlook until the end of the planning horizon.

Table 18 Industrial gas turbine market 1-20 MW size class - annual shipments (MW).

Regions	1995	2000	2005	2010
United States	330	380	450	450
Canada	120	130	140	140
Latin America	210	300	350	350
Europe	600	610	600	600
Middle East/Africa	370	410	450	500
Japan	150	220	260	280
Pacific/Asia	545	650	730	800
Total	2325	2700	2980	3120

Task 4. Energy Savings, Environmental Impact

Energy Savings

The savings of natural gas in world-wide markets that will result from introduction of ceramic hot section engines introduced in year 2003 upto 2015 can be calculated. This analysis will include first, second and third generation engine designs. Also, an assumption is made that predicted electrical needs will be met by necessary number of metallic and ceramic engines operating for 8,000 hours per year.

The world-wide savings of natural gas will be over 500 BCF per year by 2015 out of a projected world-wide consumption of somewhat over 80 TCF. If it is assumed that

35% of the installed capacity is in the USA, then the domestic savings will be over 200 BCF out of a projected domestic gas consumption of over 20 TCF. These savings may be overstated to some extent.

Environmental Benefits

The approach used is the same as for the energy savings assumptions. The reductions in emissions considered are CO₂ (greenhouse effect) and nitrous oxide (NO_x).

The market projections employed in Table 18 for gas turbines in 1-20 MW size range when compared for carbon dioxide emissions with coal-fired utility show the following.

Table 18.a. Comparison of CO₂ emissions for coal-fired utility plus gas fired boiler with ceramic hot-section mars 100 engines to meet market demands in 2005-2015.

Year	GW Demand (Cumulative)	No. of Ceramic Mars 100 Required	Steam Produced by Fleet (Million PPH)	CO Produced (Million Tons/Year)	
				Ceramic Mars 100	Utility + Boiler
2005	8	581	26	34.6	78.6
2010	24	1743	78	103.9	235.7
2015	40	2906	130	173.2	392.9

Reductions in emissions of NO_x has both environmental benefit as well as a major benefit to the OEM in commercialization. The assumptions follow the engine penetration per Table 18; the average emission estimate for conventional system is 100 ppmv NO_x at 15% oxygen; gas flow in Mars engines and the ceramic derivative is 80 lbs. per second and simple cycle used for calculations and Mars 100 ceramic hot section systems generate 8 ppmv of NO_x.

Table 18.b. Emissions of NO_x in k tons per year for ceramic fleets to meet projected electrical demand.

Year	GW Demand (Cumulative)	Ceramic Mars 100	
		Number Needed	Emissions, kTons/Year
2005	8	571	8.6
2010	24	1714	25.9
2015	40	2856	43.1

Task 5. Design Studies

Combustor Liner Applications Stress Analysis

A simplified one dimensional heat transfer analyses was performed on each of three combustor configurations to determine the stresses on the DuPont Lanxide DIMOX® Nicalon Fiber Reinforced Alumina ceramic. The three combustors analyzed were the Ceramic Stationary Gas Turbine (CSGT) lean premix, rich/lean, and catalytic configurations.

The stress analysis is an order of magnitude calculation to determine the principal stresses in the ceramic combustor can. The analysis first determines the heat transfer in the combustor and calculates the temperature difference across the ceramic combustor can in the radial direction. This temperature gradient is assumed linear for this analysis and the equations for the thermal stresses from internal constraints are found in Roark and young's Formulas for Stress and Strain, Fifth edition, McGraw Hill Book Company, page 585). The formula for a long hollow cylinder with thin walls is

$$\sigma = \frac{\Delta T \alpha E}{2(1-u)}$$

Where: σ = stress value
 ΔT = the temperature difference across liner from inside to outside
 α = the coefficient of thermal expansion
 E = Young's modulus
 u = Poisson's ration

The radial gradient across the wall produces stresses both circumferentially (around cylinder) and longitudinally (along the axis of the cylinder). Both of these stresses are found by the above equation. The stress at the inner hotter surface is compression and at the outer cooler surface tension. In addition, the stresses could be up to 25% higher if the ends are free than the values given. A summary of the results is listed in Table 18.c.

Table 18.c. Results of one-dimensional stress analysis for combustor applications.

Combustor Configuration	Temperature Difference Across Walls (°F)	Compressive Stress (psi)	Tensile Stress (psi)
CSGT Lean Premix	121	5338	3992
Catalytic	76	3328	2489
Rich-Lean	494	21706	16166

The tensile and compressive stresses are different because the tensile and compressive values for Young's modulus (E in the above equation) are different. The data used for the calculations were based upon 2000°F data for the DuPont Lanxide material as summarized below. The compressive stress occurs at the hot inner wall in both the longitudinal and circumferential directions and the tensile stress occurs at the cooler outer wall in both the circumferential and longitudinal directions. Other stresses caused by the longitudinal or circumferential temperature variations are not simple to calculate and must be done using a finite difference analysis program such as ANSYS. Unless the end constraints are very unusual, the radial temperature gradient is probably the main source for stress with some lesser contributions from the circumferential and longitudinal temperature gradients.

Coefficient of thermal expansion = 3.01 microin/inF

Young's modulus (compression) = 21.9 Msi

Young's modulus (tension) = 16.38 Msi

Thermal conductivity = 3.21 BTU/hr/ft/F

First Stage Nozzle Stress Analysis

Finite Element Analysis (FEA) was performed on a modified CSGT nozzle configuration. The material properties database that was supplied by DLC and used in the analysis is given in Tables 18.d, e, f, and g.

Table 18.d. Structural properties of DLC's DIMOX® Nicalon reinforced alumina composite.

Temp. °F	E1, E2 MSI Tensile	E1, E2 MSI Compressive	E3 MSI	a1, a2 uin/in °F	a3 uin/in °F	G23, G31 MSI	u12,u21	u31,u32
70	12.42	21.90	11.08	2.12	2.74	4.4	0.25	0.32
1000	14.40		13.62	2.56	5.76	5.5	0.25	0.28
1800	15.95		15.55	2.94	5.96	6.2	0.25	0.27
2000	16.38		16.17	3.01		6.5	0.25	0.26
2200	13.86		13.66	3.06		5.5	0.25	0.25
2400	13.80		13.76			5.5	0.25	0.25
2600	13.77		13.76			5.5		

Table 18.e. Strain to failure properties of DLC's DIMOX® Nicalon reinforced alumina composite.

Temperature °F	Xet, Yet %	Xec, Yec %	Xu, Yu crack e %
70	0.81	0.44	0.04
1000	0.70		0.04
1800	0.62		0.05
2000	0.60		0.05
2200	0.59		0.07
2400	0.63		0.07
2600	0.67		0.07

Table 18.f. Strength properties of DLC's DIMOX® Nicalon reinforced alumina composite.

Temperature °F	Xt, Yt ksi	Xc, Yc ksi	Flexure ksi	Interlaminar Tensile, ksi	Interlaminar Shear, ksi	Xu, Yu crack (ksi)
70	38.3	93.6	67	>>1.15	1.87	6.1
1000	37.5		68		1.90	7.5
1800	37.0		69		1.90	8.5
2000	36.7		70		1.90	9.1
2200	38.3		71	>3.8 2x1LS	1.91	7.8
2400	34.0		58			9.5
2600	32.7		49			9.8

Table 18.g. Thermal properties of DLC's DIMOX® Nicalon reinforced alumina composite.

Temperature °F	K1, K2 Btu/hr ft °F	K3 Btu/hr ft °F	Cp Btu/lbm °F	ε
70	5.08	5.02	0.17	0.9 0.85
1000	4.52	4.50	0.268	
1800	3.31	3.29	0.299	
2000	3.25	3.21	0.306	
2200	3.25	3.19	0.313	
2400	3.17	3.09	0.327	
2600	2.79	2.75	0.33	

The finite element model was built from 5784 solid elements with 9279 nodes. Thermal convective boundary conditions were applied to 4 surfaces. The thickness of each ply in the layup of the nozzle was assumed to be 0.015 inch. As shown by the model, there is a thermal gradient in the nozzle of about 750°F.

Recommendations from design for improving the results include:

- Separating the airfoil from the shroud
- Axial Cutting the Airfoil
- Film Cooling

Summary and Conclusions

The use of DuPont Lanxide's DIMOX™ directed metal oxidation process Nicalon™ reinforced alumina composite in three combustor components and a first stage nozzle was studied. An impact study was completed in terms of performance, energy savings, economic benefits, strategic factors, and environmental effects. Each component application was defined, and preliminary stress analysis was performed. The conclusions from this work are as follows:

1. The use of ceramics in a gas turbine hot section allows an increase in the TRIT and corresponding increases in electrical output and electrical thermal efficiency.
2. Reliability, Availability, and Maintainability are important buying criteria in the 1990's and will continue to be important after 2000. In the future, efficiency and emissions will become more important than first cost.

3. Due to their greater efficiency, ceramic hot section gas turbines would use only 83% of the natural gas that would be required by metallic hot section gas turbines to produce the same amount of energy, giving a savings of 17%.
4. Ceramic hot section gas turbines create significantly less CO₂ and NO_x than metallic hot section gas turbines.
5. CFCCs do not use significant quantities of strategic elements.
6. The use of the DuPont Lanxide DIMOXTM NicalonTM reinforced alumina composite is not recommended in the first stage nozzle at this time. Extensive redesign and improvement in material properties will be required before this CFCC can be used for the nozzle application.
7. The definition and stress analysis have shown that the DuPont Lanxide DIMOXTM NicalonTM reinforced alumina composite is a viable candidate for the combustor hot wall applications.

9.0 FOSTER WHEELER DEVELOPMENT CORPORATION

Task 1.1

Industrial Applications and Benefits

The objective of this task is to identify opportunities for industrial applications of CFCC components and select the best for further evaluation. The applications considered were:

- High-temperature heat exchangers
- Air preheater
- Steam reformers
- Miscellaneous applications
 - Processing of hazardous and toxic wastes
 - Tube supports in high-temperature fired heaters
 - High-temperature cyclones
 - High-temperature valves
 - Structural division walls

High-Temperature CFCC Heat Exchangers

The efficiency of a conventional coal-fired power plant is approximately 35%. With the continued development of high-temperature gas turbines, it is possible to substantially improve today's power plant efficiencies by means of a high-temperature, combined Brayton/Rankine cycle, in which the conventional steam cycle is topped with a Brayton cycle using an externally-fired gas turbine, refer to Figure 61.

An advanced combined-cycle plant incorporating a CFCC heat exchanger (CHX) between the conventional metal heat exchanger (MHX), refer to Figure 62, and the topping combustor would allow an increase in plant efficiency to 47%. Such advanced combined-cycle power plant will generate 34% more power per ton of coal.

Figure 61. Combined-cycle plant flow diagram.

Figure 62. Combined cycle plant with CFCC heat exchanger.

A 47% plant efficiency can be achieved with a CHX exit air temperature of 2000°F and a natural gas contribution of approximately 26%. As improved CFCC materials are developed in the future, it will be feasible to heat air in the CHX to 2300°F or higher, thus completely eliminating the need for a natural gas topping combustor.

An increase to 47% total plant efficiency would decrease airborne emissions (CO, NO_x, SO_x and particulates), and total solid wastes (coal ash and wastes from the flue gas treatment system) by at least 25%, and decrease thermal loadings to cooling water by at least 33%.

The higher efficiency also yields environmental benefits throughout the entire fuel cycle, reducing coal mining and transportation requirements. Reduction in emissions of particulate matter, sulfur, carbon, and nitrogen oxides, will help reduce the potential adverse environmental impact of these emissions — acid rain and greenhouse effect (global warming).

Market Potential For High-Temperature CHX

New electricity generating plants will be needed to meet continuing increases in demand for electric power. In addition, a large number of existing generating units will reach the end of their useful service lives and new units will be required to replace this existing capacity.

Today, the U.S. has approximately 780,000 MW of installed electric generating capacity. Assuming moderate growth rates of 2% annually and including replacement of older units, it is estimated that about 200,00 MW of new capacity must be installed before the year 2000. Since coal is the nation's most abundant energy source, it is reasonable to assume that a large share of this new capacity will be derived from coal-based combined gas turbine/steam turbine plants.

World installed electric capacity is approximately four times that in the U.S. its projected growth is conservatively estimated at 2% per year, which translates to over 500,000 MW of new capacity required by the year 2000.

Heat Pipe CFCC Air Preheater

The efficiency of power generation plants can also be increased by lowering the exhaust flue gas temperature from the boiler furnace used in Rankine (steam) power cycles.

Cycle efficiency calculations show that by lowering the stack gas temperature by 100°F, the plant cycle efficiency can be improved by approximately 1%.

Such a power plant will generate approximately 2.8% more power per ton of coal, decreasing airborne emissions (CO, NO_x, SO_x and particulates) and total solid wastes (coal ash and wastes from the flue gas treatment system). The potential market for CFCC air preheaters includes not only the power plants that must be installed to fill the new capacity required by the year 2000, but also the retrofit of old plants that must be revamped to meet new pollution standards.

CFCC Steam/Methane Reformers

Steam reforming of natural gas is a process in which steam and methane are used to produce synthesis gas (mainly a mixture of hydrogen, carbon monoxide and steam). Depending upon the application, the synthesis gas can be used downstream of the reformer to produce methanol, hydrogen or ammonia as the final product.

A typical steam/methane reformer consists of a radiant furnace-type section with parallel reforming tubes in the firebox, and a convention section which recovers the furnace flue gas heat, refer to Figure 62.a.

Figure 62.a.

The steam reforming reaction in the reformer tubes is carried out at relatively high temperatures and pressures. The stresses generated in the tubes limit the inside reforming temperatures (reactant temperatures). In current state-of-the-art reformers, the maximum reaction temperatures and pressures inside the tubes are limited to about 1450°F and 500 psig for ammonia plants, and to about 1600°F and 300 psig for methanol plants.

The process efficiency during conversion of methane to synthesis gas increases with temperature. Current state-of-the-art reformers using metallic tubes operating at 1600°F are limited to about 86% conversion of hydrocarbon to synthesis gas. Using CFCC tubes in the reformer, the reaction temperature inside the tube could be increased to about 1900°F, which will increase hydrocarbon conversion to about 99%.

For example, steam reforming using CFCC tubes has the potential to save up to 19% in feedstock requirements and up to 30% in total energy requirements compared with the same size methanol plant equipped with a conventional reformer. These savings are directly attributable to the higher reforming temperatures and pressures that could be possible with the use of CFCC materials.

For a typical methanol plant, the energy savings are estimated at 8.3×10^6 Btu/ton of methanol. For a 1000 TPD methanol plant with 90% availability, this is equivalent to 2.73×10^{12} Btu/year.

Market Potential for CFCC Reformers

The market potential for steam reformers depends to a large extent on the demand for clean liquid fuels. Federal regulations have the potential to at least double the U.S. demand for methanol within 10 years. Similar increases are expected in Europe and Japan. The current world capacity for methanol production (about 9 billion gallons per year) is well below the projected level of consumption. Therefore, considerable new capacity will be required, both domestically and overseas.

Conservative estimates project that U.S. demand for methanol will increase from a current 1 billion gal/yr (bg/y) to 6 bg/y within 10 years through a combination of methanol-fueled vehicles, increased use of MTBE and other uses. Assuming that 25%

of the new capacity is built in the U.S., approximately 10 new 1000 TPD methanol plants would be required in this country alone. The potential in foreign markets is even greater. Assuming CFCC reforming technology captures a 25% share of the new methanol plant construction, 24 CFCC steam reformers would be required worldwide.

Task 1.2

Assessment of Candidate Industrial Applications

The candidate industrial applications described earlier were assessed regarding potential energy savings, environmental benefits and cost effects of CFCC technology. The following screening/evaluation criteria were used:

- Energy savings (effect on plant efficiency)
- Environmental benefits (effect on pollution emissions)
- Feasibility
- Cost
- Market potential

An assessment of candidate applications is summarized below:

High-Temperature CHX

- Potential for significant increase in power plant efficiency. Total potential annual energy savings of 0.5 quad/year (500×10^{12} Btu).
- Potential for significant reduction in pollution emissions resulting from decreased fuel consumption (higher efficiency).
- Moderate technical risk and development costs. Technical performance requirements for CHX are in Table 19.

- Plant capital and operating and maintenance (O&M) costs are expected to be higher but higher cycle efficiencies will result in significant fuel costs savings. Total fuel costs to the power industry could be reduced by 13% (\$1.3 billion/year).
- Potential for large market in new installations over the next 10 years.

Table 19. Technical performance requirements.

CFCC Heat Exchanger (CHX)

Parameter	Tubes*	Headers*
Outer Diameter	2 to 3 in.	Up to 8 in.
Wall Thickness	1/16 to 1/4 in.	1/4 to 1/2 in.
Length	10 to 30 ft.	Up to 10 ft.
Max. Surface Temperature	2200 to 2300°F	Same as Tubes
Max. Pressure Differential	200 to 250 psi	Same as Tubes
Air Inlet Temperature	1400°F	Same as Tubes
Air Outlet Temperature	1800 to 1900°F	Same as Tubes
Air Side Pressure Drop	5 psi	—
Flue Gas Temperature	3300°F (max.)	—
Flue Gas Pressure	Near atmospheric	—
Flue Gas Velocity	80 to 150 ft/sec	—
Service Life	10 years	Same as Tubes
Heat Transfer Coefficients:		
Air Side	10 to 20 Btu/h-ft ² -°F	
Flue Gas	50 to 100 Btu/h-ft ² -°F	

* The CFCC material tubes and headers will be required to have structurally sound joints. One possible air heater configuration will require U-tube arrangement where the 2- to 3-in. diameter tubes will have 180° bends with 2- to 6-in. radius.

CFCC Reformers

- Potential for significant improvement in process efficiency and reduced energy requirements. Total potential annual energy savings of 0.25 quad/year.
- Potential for significant environmental benefits from the end product (use of methanol, hydrogen and other reforming end-products to produce cleaner fuels and reduce pollution from automobiles).
- Moderate-to-high technical risk and development costs. Refer to Table 20 for CFCC targeted properties.
- Plant capital cost is expected to be higher. O&M cost are expected to be lower because higher process efficiencies and reduced energy requirements will result in significant fuel and feedstock costs savings.
- Potential for a very large market for methanol production (demand for methanol expected to increase significantly over the next 10 years); additional smaller potential market for ammonia and hydrogen production.

CFCC Air Preheater

- Small (1%) increase in power plant efficiency.
- Small reduction in pollution emissions.
- Moderate technical risk and development costs. Feasibility of a ceramic heat pipe uncertain because of leakage concerns.
- Operating costs expected to be lower but maintenance costs would be higher than those for a conventional power plant. Plant capital cost expected to be higher.

- Potential for large market in new installations and retrofit of plants now in operation.

Targeted Properties for CFCC Tubes and Headers

- High-temperature strength
- Good thermal conductivity
- Low thermal expansion
- High thermal shock resistance (600°F in 5 min.)
- Oxidation resistance
- Corrosion resistance
- Resistance to alkali attack
- Creep strength
- Slow crack growth
- Low permeability
- Erosion resistance to coal ash

Table 20. CFCC target properties for HTAH and steam reformer.

Property	Criterion	Desired	Required
Strength at Operational Temperature (psi)	Stress at Initiation of Matrix Cracking	10,000	6,000
Creep Rupture Stress (psi)	Operating Temperature and Design Life	10,000	6,000
Thermal Conductivity (Btu/hr-ft°F)	Tube Through-thickness Conductivity	>6	>5
Coefficient of Thermal Expansion ($\times 10^{-6}$ in/in/°F)	Normal Operation Temperature Gradients. Upset Conditions.	3	<5
Thermal Shock (°F/min)	Upset Conditions (600°F Ramp)	120	120
Strength Degradation Due to Environment Attack (%)	Stress at Matrix Cracking Initiation	0	5
Permeability	Operating Conditions	0	0
Erosion Resistance	Coal Ash for HTAH (10 Micron)	0	0

Task 1.2.3

Economic Targets for CFCC Air Heater

Economic evaluation and identification of economic targets for CFCC Air Heater are closely related to the power generation system in which it is used as well as efficiencies and emission controls of the system. A high performance power system (HIPPS) cycle that FWDC is developing under contract from the Department of Energy has been selected as an ideal candidate for the CFCC air heater. The CFCC air heater is designed to heat 2,960,000 lb/hr of air from 1400 to 1800°F in the HITAF furnace, using 2-inch diameter CFCC tubes.

The total cost of the air heater is very sensitive to the cost of CFCC tubes. The current price of the 2-inch CFCC tubes based on the low experimental production levels is very high. Thus for estimating purposes we have parametrically varied the price of the CFCC tubes from \$50 to \$200 per foot.

The price of CFCC air heater would be \$10.15 million if the price of the tubing is \$50 per foot. The price of comparable monolithic ceramic heat exchanger was estimated to be \$6.78 million in a parallel study being conducted by FWDC under contract from DOE. Thus the CFCC heat exchanger is about 50% more expensive than the monolithic ceramic heat exchanger.

The increased availability and decreased maintenance costs of CFCC air heaters compared to monolithic ceramic air heaters as well as gains due to reduced forced outages will more than offset the 50% higher cost of CFCC air heaters. However, for CFCC tube costs higher than \$50/ft, the use of a CFCC air heater in the HIPPS system will begin to be economically unattractive.

In view of these considerations, it is our judgement that if the cost of the CFCC tubes can be reduced to the level of \$50 per foot or below, the CFCC air heaters will be economically viable in the HIPPS System.

Economic Targets for CFCC Steam Reformer

The low-pressure all metal conventional hydrogen plant and a high-pressure hydrogen plant, using a CFCC Steam Reformer are rated to produce 50 MMSCFD hydrogen at 950 psig for downstream processing.

A cost analysis indicates total capital costs for the conventional and the high-pressure plants (CFCC reformer) to be approximately 40 and 30 million dollars, respectively. The latter cost does not include the cost of the CFCC reformer.

The design of a two-stage reformer for application in the high-pressure hydrogen plant uses metallic tubes in low-temperature region of the reformer furnace and CFCC tubes in the high-temperature region. The design incorporates a total of 264 3.0 inch I.D. x 0.375 inch wall thickness tubes. Each tube is 44.5 ft. long and consists of a 22.5 ft. metallic section and a 22 ft long lower CFCC section.

Supplementing the cost of the high-pressure hydrogen plant with the CFCC reformer, the total capital cost for the high-pressure plant, excluding cost of the CFCC tubes, is 40 million dollars. This is the same as the total capital cost of the conventional low-pressure hydrogen plant. This indicates that the capital cost of the high-pressure plant using a CFCC reformer will exceed the capital cost of the low-pressure conventional plant by the cost of the CFCC tubes.

There is a 1.3 million dollar annual operating cost advantage for the high-pressure plant over the low-pressure conventional plant. We believe that the addition to the capital cost of the high-pressure case due to CFCC tubes should be recoverable in one or two years. Assuming constant dollars, this implies 1.3 to 2.6 million dollar allowance, for the CFCC tubes in the high-pressure case. After the second year, the high-pressure plant will provide a 1.3 million dollar annual cost savings.

The 1.3 to 2.6 million dollar capital cost allowances for the 264 CFCC tubes (3 inch ID x 0.375 inch wall x 22 ft. long) translates into per tube cost of approximately \$5,000 and \$10,000, respectively. We believe the development of the CFCC tubes for high-pressure reformer application should be targeted for this price range.

It should be noted that while the cost estimate for the conventional low-pressure hydrogen plant was based on actual plant data, a conservative design and cost estimating approach was used for the high-pressure plant since no such plant currently exists. We believe the high-pressure plant will be more cost competitive if the design and cost estimating were performed in more detail. This was not possible in the current effort.

Also, we believe the reliability of the CFCC tubes at the required service conditions should be higher than that for the metallic tubes. In fact, currently available metallic tubes can not be used at such high pressures and temperatures.

Task 1.3 Design Studies

CFCC Air Heater

The CFCC Air Heater (CAH) is designed for heating air from 1400 to 1800°F in the High-Temperature Advanced Furnace (HITAF), as part of the 290-MWe High Performance Power System (HIPPS). A simplified schematic of the HIPPS plant is shown in Figure 62.b. and a schematic of the CAH is shown in Figure 62.c. Pulverized coal and sorbent are fed to the pyrolyzer, where the coal is partially gasified, producing a low-Btu fuel gas and combustible char. The fuel gas is then fired in sections of the HITAF where ash deposition and corrosion would be a problem. The char is also fired in the HITAF, but the design of the HITAF is such that the products from the char combustion are present only in areas of the boiler where ash deposition can be tolerated and controlled.

There are two potential situations that can cause corrosion of CFCC materials. One is at tube temperatures of around 2200°F, where many types of ash will be molten, and the alkalis in the ash will react with the CFCC tube material. The other region of concern is around 1800°F. At this temperature alkalis are condensed from the flue gas, greatly increasing the alkali concentration on the tubes.

The HIPPS concept solves both corrosion problems by cooling and filtering the fuel gas before it is fired in the fuel gas furnace. After leaving the secondary cyclone, the fuel gas is cooled to 1200°F in a heat exchanger. At this temperature most of the alkalis in the fuel gas will condense. The condensed alkalis and the particulates that escape with the gas from the secondary cyclone are then removed in a barrier filter.

The fuel gas leaving the barrier filter is then sufficiently clean to prevent ash deposition and corrosion problems in the CAH.

Figure 62.b. CFCC Air Heater (CAH) application in HITAF (HIPPS).

Figure 62.c. Proposed heat exchanger design.

CFCC Steam Reformer

In our initial work, we considered a heat exchange steam methane reformer/partial oxidation (SMR/POX) combination to maximize the advantages of CFCC properties. The SMR/POX plant was designed to produce a synthesis gas suitable for use in a methanol synthesis loop. This scheme was chosen since the combination of reforming and partial oxidation can produce a syngas with the H_2/CO ratio suitable for methanol synthesis. A detailed analysis, however, revealed that an optimum SMR/POX

system did not fully utilize CFCC high-temperature strength because of the lower tube temperature and differential pressure requirements for this reformer.

Further evaluation suggested use of a CFCC reformer in a hydrogen plant that would produce high-pressure hydrogen without using a downstream compressor. This requires not only an increased pressure at the outlet of the reformer but also an increased temperature due to pressure effects on the reaction equilibrium. Thus, this case very clearly took advantage of the properties of the CFCC materials.

A standard hydrogen plant using a conventional metallic steam reformer and producing 50-MM SCFD hydrogen product gas at 351 psig was used as the reference. Since most downstream processing options in a hydrogen plant require product gas at higher pressures, system design of a 50-MM SCFD hydrogen plant delivering product gas at 940 psig was considered for a CFCC reformer application. To maintain product composition, the higher pressure requires reforming at a higher temperature, thus delivering product gas at 1815°F versus 1535°F for the conventional plant.

For the high-pressure plant, it was determined that a two-stage reformer will best utilize the high-temperature strength of the CFCC material. The first stage will use metallic tubes and produce synthesis gas at 1030 psig and 1435°F. These conditions approach the strength limits of the conventional metallic tubes. The second stage uses CFCC tubes yielding a final outlet synthesis gas at 1000 psig and 1815°F.

In comparing design, performance and economics of the conventional and the high-pressure hydrogen plants, a product compressor was added to the conventional case to make the outlet pressures of the product gas the same.

10.0 GENERAL ELECTRIC POWER GENERATION

Task 1 Application Assessment and Benefits

The use of high-temperature ceramic composites in the first stage turbine inner shroud and combustion liner applications can significantly reduce cooling air usage which leads to significant fuel savings and reduced emissions harmful to the environment. Benefit studies completed earlier yield very impressive results for the use of ceramic composites in the turbine shroud and combustion liner applications.

The benefit analysis was conducted for an "optimized" CFCC shroud and combustion liner design for a 7FA, 160 MW industrial gas turbine application. The cooling flow and pressure drop reductions used to quantify a benefit (fuel savings, emissions reduction) are the targeted values for the optimized component designs. Preliminary analysis results indicate these values could be met based on the high temperature capability of CFCC's.

The first stage turbine shroud is located just downstream of the first nozzle and outboard of the first stage bucket. (Ref. Fig. 63). Consequently, it is exposed to some of the highest gas temperatures in a gas turbine. CFCC materials may satisfy the shroud mechanical and environmental requirements without the need of cooling air, or at least, with a major reduction in the cooling air flow. By applying a CFCC to the first stage turbine inner shroud of the 7FA, a cooling air reduction of 1% may be realized. This approximate one percent reduction in cooling air can increase turbine power output (delta KW- 1.0 percent) and reduce heat rate (0.4%) for the same fuel consumption or

reduce fuel consumption for the same power output and heat rate. A fuel savings of \$460,000 could be realized per year for a 7FA machine with a CFCC shroud.

Gas turbine manufacturers throughout the world are currently pursuing technology for reducing the air pollutant emissions in the exhaust. The most common pollutant generated by the combustion of conventional fuels such as natural gas and No.2 distillate oil are NO_x, CO and UHC. A promising method for reducing these emissions is lean premixed combustion. In order to reduce the flame temperature in the reaction zone of a modern efficient gas turbine below the NO_x formation threshold temperature nearly all the air available to the combustor must be premixed with fuel upstream of the reaction zone. If the combustion liner is film air cooled, which is necessary with conventional technology metal super alloy liners, the fraction of the combustor air not available for premixing with the fuel is 30 to 40%. If this much air is not available to the chemical reaction, it is not possible to operate the combustor lean enough to remain below the thermal NO_x formation threshold temperature at high load. The problem of remaining below the thermal NO_x temperature threshold can be solved by the use of a CFCC combustion liner which can withstand the temperature of the hot gas in the combustor reaction zone without film air cooling. In addition to, the reduction in NO_x emissions obtained by using the available air for lean premixing rather than film air cooling, a reduction in CO and UHC emissions can also be obtained through the use of a ceramic liner. A 0.5% additional flow through the 7FA combustor, (as a result of reduced cooling to the shroud for example) at base load condition, could reduce the NO_x levels 10-25% (based on laboratory results). It is conceivable a CFCC combustion liner may be necessary to meet future emission requirement in the next century.

Figure 63. Cross section of hot gas components of an industrial gas turbine.

The aerodynamic design of a ceramic composite combustion liner will consist of developing methods of reducing emissions, (NO_x and CO). while reducing the pressure drop of the combustion system in order to increase overall cycle efficiency. The realization of a reduced pressure drop in the combustion system is achieved by reducing the aggressiveness of the backside film cooling required to keep current metallic components cool enough for long term operation. The process that would be used to reduce the pressure drop would be the opening of the diameter of the flow sleeve that encircles the combustion liner. Refer to Figure 64 for a detailed view of the combustion

system. The same mass flow of cooling air now passes through this larger channel, reducing its velocity and resulting in a reduction in the pressure drop. A lower pressure drop leads to an increase in cycle efficiency and megawatt (MW) output. By applying a ceramic composite to the combustion liner, a performance improvement may be realized in the form of reduced ΔP (1.25%) across the combustion liner. This reduction in pressure ratio increases power output (ΔkW -0.75%) and reduces the heat rate of the combustor 0.375%, or for the same power output, reduces fuel consumption and heat rate of the machine. A fuel savings of \$370,000 per year could be realized for a 7FA machine with a CFCC combustion liner.

Figure 64. Combustion system cross section.

Based on the shroud and combustor analysis, a 2% improvement in power output of a 7FA machine can lead to fuel savings of \$1 million per year. In the year 2000, based on a projected US base of 100 machines, this is expected to be \$100 million per year and for 140 machines world wide \$140 million per year. Based on the marketing data for the 7FA and given the potential benefits for an optimized CFCC shroud, the target cost of a set of ceramic shrouds for one 7FA machine may be as much as three times the cost of a set of metallic shrouds. Using similar assumptions, the target cost of a set of CFCC combustion liners for one 7FA machine may be between four to five times the cost of a set of metallic liners.

Task 1.2

Turbine Shrouds

The CFCC turbine shroud which is targeted for a retrofit application on the MS7001FA model industrial gas turbine must withstand the same operating requirements as the metallic turbine shroud it replaces. For this application, the life requirement for the CFCC shroud is 48,000 hours total operating time. During this time, the machine is expected to complete 500 normal startup and shutdown cycles. During startup and shutdown procedures, no special allowance for CFCC turbine shrouds are to be considered. In addition, 35 turbine trips or emergency load rejection cycles are expected during the same time frame. Sound design principles are required to evaluate the CFCC shroud in the turbine system. Consideration for EOD (Engine Originated Damage) must be evaluated. When subjected to specific types of EOD, the shroud must be designed not to fail catastrophically. Other considerations, beyond the scope of this program, which have the potential to limit the CFCC shroud operation are deterioration from oxidation, corrosion, erosion, creep deformation or spalling. For

maximum performance, clearances between the bucket tip and shroud should be at a minimum.

Combustion Liner

The CFCC combustion liner which is targeted for a retrofit application on the 7FA model IGT must withstand the same operating requirements as the metallic liner. The operating requirements of combustion components in an IGT for the power generation market demand that the components have a level of durability sufficiently capable of use at temperature for 24,000 hours. The firing temperature of the machine is 2350F. The combustion components are inspected every 8,000 hours to assess their condition, at which time the parts may be removed for repair and reinstallation. After three cycles of inspection and repair, the combustion liner and some of the associated components are permanently removed. Over the operating life of the combustion components, they must be able to withstand 250 normal start/stop cycles. The combustion liner must be able to operate on natural gas fuel and distillate oil fuel to assure full time availability for power generation. The combustion hardware must retain adequate mechanical properties to withstand the effects of water.

Material Systems

The CFCC composite material systems consisted of the following:

- DuPont Lanxide Composite (DLC) DIMOX®, directed metal oxidation material system.
- DuPont Lanxide Composites CVI material system.
- The Amercom CVI material system.
- GE-CRD/Textron Toughened Silcomp material system.
- The Babcock and Wilcox material system.

A summary chart of the five composite material systems evaluated is provided in Table 23.

Table 23. CFCC material systems

Vendor	Fiber	Preform	Matrix
Amercom	Nicalon (SiC)	Laminate, Weave, or Braid	CVI SiC
Babcock & Wilcox	Mitsui Al ₂ O ₃	Filament Wind	Sol-Gel Al ₂ O ₃
DuPont CVI	Nicalon (SiC)	Laminate, Weave, or Braid	CVI SiC
Ge-CRD	SCS-6 (SiC)	Laminate	Melt-Infiltration SiC
Du-Lanx Composites	Nicalon (SiC)	Laminate, Weave, or Braid	Direct Metal Oxidation (DiMox) Al ₂ O ₃

Task 1.3

Turbine Shroud Design

The two piece shroud design for the first stage is shown in Figure 65. There are 32 outer shroud segments which assemble to the turbine casing to form the 360 degree turbine annulus area. There are 96 inner shrouds (three inner shrouds per outer shroud segment) which form the outer flow path structure of the turbine annulus. As a consequence of the shroud function and location, the inner shroud surface is exposed to some of the highest gas temperatures and velocities in the gas turbine of approximately 2200F. In addition to the high temperature, the gas side surface of the shroud must resist very high gas velocities (approx. 750 ft/sec axial and 400 ft/sec tangential). The

cooling air required to maintain the metal shroud temperatures below 1800F is approx. 1 percent of the compressor mass flow. As a flow path member, the shroud must be structurally sufficient and capable of meeting the design life requirements in the given temperature and pressure environment. Both structural integrity and oxidation/corrosion resistance are prime concerns.

Figure 65. Stage 1 turbine shroud.

Eleven conceptual designs were developed during the course of this program. These conceptual designs ranged from easy CFCC manufacturing and complex metal support structures to more complex CFCC manufacturing with simpler metallic structural designs. One very important feature of the CFCC design is the interface region between the CFCC and the supporting metallic structures. A compliant layer will ensure that no localized point loading will occur and proper distribution of the contact load will be maintained. A second function of the interface is as a thermal barrier. Heat transmission from the CFCC shroud into the metallic support structure should be minimized. A third function of the interface is to prevent chemical attack of the CFCC products on the nickel based support structures. The rub considerations may drive the need for a thermal barrier coating or graded composite structure on the surface which faces the flow path. This new view of a CFCC application needs extensive development work to achieve complete success with a CFCC turbine shroud.

After review of 11 conceptual designs, three providing the most promise were selected for further study:

1. Concept "C" - Most manufacturable CFCC design.
2. Concept "G" - More complex CFCC design but less complex metallic structural design.
3. Concept "I" - More complex CFCC, lower stress levels but least complex metallic structural design.

A structural analysis of the CFCC inner shroud, 3-D ANSYS models of concepts "G" and "I" were constructed to determine temperature, thermal stress and mechanical stress distributions in the component. Concept "I" had slightly lower stress levels and

higher margins of safety than concept "G". Program funding and timing did not allow an ANSYS model of concept "C" to be constructed and analyzed. This CFCC component would be expected to have the lowest stress levels of all three designs. However, complex metallic support structures may prohibit use of this design. A more thorough investigation of this concept should be conducted in Phase II. Further design studies are required to quantify the amounts of cooling air reduction possible with each material system.

Overall, it is considered possible to design with all material systems reviewed. Proceeding from the initial assumption of a laminate fiber architecture and pursuing a more complicated 3-D fiber architecture would probably make the CVI systems more adaptable to the design. Additional optimization of the densification process would help in all the systems.

It must be kept in mind that this preliminary study can only identify trends. A detail design must be completed for each material system. This would have many variables which would be looked at to determine a viable turbine shroud system incorporating a CFCC inner shroud. Also, this analysis does not consider the environmental issues associated with these material systems. Very little data, if any, was provided to determine a life prediction for these material systems. Once that data is acquired, a formal life assessment can be made. A life methodology and prediction may be possible only near the end of Phase II.

Figure 68. **Concept "T" - circumferentially pinned support shroud.**

Figure 69. **Concept "C" - wedge support shroud.**

Figure 70. Concept "G" - axially pinned support shroud.

Combustion Liner Design

The 7FA industrial gas turbine consists of fourteen of these assemblies located circumferentially around the machine. All combustion components are stationary with the exception of the retractable spark plug for ignition. The combustion liner is constrained at both ends, specifically by a seal on the tail-end and by stops on the head-end. The mechanical loads experienced by the combustion liner during operation are thermal stresses due to temperature gradient and a compressive load associated with the pressure drop across the liner. The pressure drop the liner experiences is an effect of the pressure losses that occur within the combustion system to provide cooling and introduce the air in combustion reaction. Air is exhausted from the compressor discharge casing into the combustion wrapper at a temperature above 700F and a pressure near 15 atm during base-load operation. Elimination of film cooling is the single most effective way of reducing emissions, but this has a negative effect on temperature operating limits of a metallic combustion liner. The elimination of film cooling could raise the temperature from 1500F to 1700F. Increasing the pressure drop to gain more effective cooling can be done to overcome this component temperature increase. However, a loss to overall efficiency of the gas turbine results.

In generating the basic geometry of the initial design for a ceramic combustion liner, it became evident that the manufacturing of a component this large could be extremely difficult and expensive. In addition, many of the composite manufacturers do not possess the furnace size capability to fabricate a 16 inch diameter by 36 inch length part on a production basis. A segmented design may prove challenging due to the many joints to be sealed. However, the small tiles will be easier to manufacture and have higher reliability in meeting the necessary design requirements. Many combustion

systems have circumferential temperature variations associated with the unevenness of fuel distribution within the combustion chamber. These hot streaks would have a significant effect on the thermal stresses of the component. The development of a cylindrical ceramic composite combustion liner may not be able to withstand the thermal stress fields generated by hot streaks.

The segmented design of a ceramic composite combustion liner was focused on two specific geometries: one being the stacked ring (Figure 66) and the other being tiles arranged both circumferentially and axially to form a combustion cylinder (Figure 67). Again, the effect of hot streaks was considered for each geometry. The stacked ring design would be effective in eliminating the axial thermal gradient concern, since the rings would be susceptible to the circumferential thermal gradients that a full-length cylindrical component experiences in a land-based gas turbine. The tile concept for making a combustion liner will allow the individual tiles to react independently of each other, therefore minimizing thermal stresses associated with the axial and circumferential directions. The sealing concept envisioned for the tiles attached to the metallic substructure will limit the leakage of backside cooling air into the combustion chamber while also providing a compliant interface to lessen the effect of thermal expansion disparities between metal and ceramic.

Figure 66. **Stacked ring combustion liner.**

Figure 67. **Segmented tile combustion liner.**

The four major categories in the material evaluation matrix of Design, Material Properties, Manufacturing and Cost tend to drive the geometry of a ceramic composite combustion liner to be an assembled tile configuration. The design aspects of a tiled configuration are not the clearest and a case could possibly be built to substantiate a cylindrical concept. The complexity of assembly and sealing a tiled configuration for turbine operation are essentially unknown. The cylinder would be inherently easier to assembly with a marked reduction in surfaces to seal. However, if a cylinder is to become a reality, the material properties of the composite systems would have to be greatly improved. The current material properties are within range of allowing a tiled configuration to meet many of the expected design requirements for combustion applications with sealing being the major challenge. Table 24 summarizes the manufacturing drivers that contributed to selection of the tile configuration concept. Although there exist unknowns related to the viability of a tiled combustion liner configuration, the merits of this design appear to be greater than a large one-piece cylinder, given the current status of the material systems investigated.

Table 24. Component manufacturability relative costs.

Rank Cost	Full Cylinder	Segmented Rings	Shingled Panel
Tooling	Highest	Intermediate	Lowest
Fiber Preforming	Complex; Automated	Complex; Automated	Simple; Manual
Matrix Densification	High	High	Lowest
Post Machining	Equal	Equal	Equal

Thermal and structural analyses were performed on the above designs using ANSYS Engineering Analysis System. Base load operating conditions were used to model the component designs, since this is the highest temperature and pressure condition seen during normal steady load operation. Table 25 shows the average bulk temperature for each material system. It is interesting to note that the composite material systems with a higher thermal conductivity have a larger increase in their bulk temperature with the reduction in cooling efficiency. This will have a more pronounced effect on the long term material property limits.

Table 25 Combustion tile average bulk temperatures.

Composite System	Current Combustion System	Reduced Pressure Drop
DLC CVI	1533 F	1656 F
GE-CRD/Textron	1597 F	1722 F
DLC DIMOX	1625 F	1725 F

For combustion liner applications, ceramic composites are well suited to provide the necessary increase in gas turbine cycle efficiency by reducing the aggressiveness of the cooling circuit. A generic design has shown that these materials have good feasibility of operating in this environment. The design did reveal some short comings of the composite materials, but none that could not be overcome by improvements to the composite system and/or modifications to the design. All of the materials evaluated in the thermal and structural analyses can be made to work in the application.

Evaluation of Material Systems For Industrial Gas Turbine

A list of categories was generated, under which each material system would be evaluated. As the list grew to 19 items, it was grouped into four major categories: design, material properties and database, manufacturing, and cost. A detailed explanation of the 19 subcategories is given in Table 26. It was decided that the 19 items were not of equal value (e.g. meeting design life requirements was more important than initial component cost). Each of the 19 subcategories was then assigned a weight factor, 3, 2, 1, according to the importance level, decided by the PGE team, of that subcategory. The weights are noted in Table 27.

When the weights of the 19 categories are summed into their 4 respective categories, the percentage of each major category to the total is:

<u>Category</u>	<u>% of Total</u>
Design	33.3
Materials	25.6
Manufacturing	28.2
Cost	12.8

Based on these four categories the DLC DIMOX® material system received the most favorable placing.

It should be made clear, the composite systems evaluated in this report are not at the same level of maturity. Given the difficulty to predict future material advancements for each of the suppliers, the evaluation team focused on "today's status" of the material system development. Therefore the ranking presented represents "a snapshot in time".

Table 26. Evaluation matrix subcategory description.

The 19 categories of the evaluation matrix and a general rationale for each are as follows:

- A. Optimum Architecture Achievable - is the fiber preform capable of having the designed architecture.
- B. Design Life - what is the capability of the material system in meeting turbine design life requirements.
- C. Material Property Requirements - does the composite system have the ability to meet minimum strength requirements and does the designer feel confident in the data.
- D. Fits Turbine System - can the composite system be integrated into the current turbine envelope.
- E. Maintainability - accessibility of the composite or component design for overhaul and inspection.
- F. Repairability - ability of composite design and assembly to allow repair.
- G. Database Quality and Quantity - amount of measured and estimated materials properties and consistency thereof.
- H. Repeatability - is the fabrication process mature or consistent enough to yield consistent property data - that will give the designer confidence.
- I. Mechanical Property Testing Capability - does the vendor have test facilities available and is the vendor capable of making the required test coupons.
- J. Environmental Stability - will the composite system be chemically stable at the turbine operating conditions for the entire design life.
- K. Experience - does the ceramic vendor have development and production manufacturing background experience, especially in composites, that will enable timely introduction of components with high quality.
- L. Preform - does the vendor have the ability to make the preform to the desired geometry.
- M. Fiber - this is the primary reinforcement in the composite system. Capabilities, including properties, preform processing, and handling ease of the fiber.
- N. Matrix Processing - what are the capabilities and properties of the matrix, and the method of composite densification.
- O. Production Scale-Up Capability - what are the capabilities of proceeding in transfer from development to production.
- P. Component Cost - what is the estimated relative cost to manufacture the specific component.
- Q. System Cost - what is the estimated relative cost or impact that the specific component has to surrounding components.
- R. Design Cost - what is the estimated relative cost to design a component with this composite system.
- S. Machine Cost - what is the estimated relative cost of this component on the total overall cost of the machine?

Table 27. Evaluation matrix subcategory weights.

Category	Subcategories	Weight
Design	Optimum architecture	2
	Design life	3
	Material Property requirements	2
	Fits Turbine system	3
	Maintainability	2
	Repairability	1
Materials	Database quality and quantity	3
	Repeatability	3
	Mechanical property testing capability	1
	Environmental stability	3
Manufacturing	Experience	3
	Preform	2
	Fiber	2
	Matrix processing	1
	Production scale-up capability	3
Cost	Component cost	1
	System cost	2
	Design cost	1
	Machine cost	1

Phase II Recommendations

In Phase II, component designs will be defined, manufactured and tested. This requires a significant design effort with close attention paid to system issues. It is important to select a configuration that can be manufactured, mate properly with neighboring hardware, and survive the extreme conditions of a gas turbine operating environment. Some aspects of designing with ceramic composites are only just beginning to be understood. Substantiation/verification and sharing information in this area will be worthwhile.

Integration of the desired fiber architecture with the preform manufacturing techniques should be given a high priority. Design and manufacturing cannot work independently of each other.

Once the preform can be manufactured, integration of the fiber preform with the densification process must be coordinated.

Development of a material database that is consistent with desired material properties based on component design requirements is critical. The results in Phase I identified the material properties which need the most development work and address the weakness of each material system.

Given that the properties which require the most development have been identified, a meaningful materials test program which identifies unique testing techniques and provides the most cost effective solution to data gathering are required.

In Phase II, prototype components will be manufactured. It is important to conduct meaningful rig tests. These tests will identify current strength levels, provide justification for process improvements, and provide the direction for future materials development.

Cost estimates for production components should be conducted in Phase II. Economic justification is always required and should be addressed.

The long range objective of this program is to conduct a full scale machine test. Component design and rig tests should simulate actual running conditions as much as possible and address the key area of concern to achieve a successful machine test program. Before any machine test will occur, a significant amount of additional amount of material property test data will be required.

Material Supplier Recommendation

DuPont Lanxide Composites - CVI

Advantages - Good start in developing the database necessary to design CFCC components. Future material development should provide a material which could survive in a turbine shroud application.

Manufacturing capabilities are at production readiness. Ready to launch a full scale development program aimed at a commercial application.

Disadvantages - The CVI process has an inherent disadvantage with closed porosity. This leads to lower micro-crack yield strengths and susceptible to environmental attack. A more comprehensive process review and alternative approach to environmental protection is required. Future materials development techniques already in place should minimize these concerns.

Material database needs more component specific data collection. Properties identified as limiting in the stress analysis should be given top priority in Phase II. Longer term material properties data for environmental effects is needed.

DuPont Lanxide Composites - DIMOX®

Advantages - Excellent production facility and manufacturing process provides flexibility with fiber architecture and component geometry.

Good start in developing the database necessary to design CFCC components. Future material development should provide a material which could survive in a turbine application.

Disadvantages - Current material system has micro-cracking matrix problems. This leads to lower yield strength and susceptibility to environmental attack. Need to develop the material system further. Micro-cracking matrix problems appear solvable with 3-D fiber reinforcement or alternate matrix composition approaches. This will require considerable material development.

Material database needs more component specific data collection. Properties identified as limiting in the stress analysis should be given top priority in Phase II. Longer term material properties data for environmental factors is needed.

Excess aluminum in matrix may bleed out during engine operation. Most of it can be removed chemically, but not all. This residual aluminum, however, may act as a matrix crack healer during operation.

Market Potential and Commercialization

Application of CFCC combustion liners and shrouds to gas turbines has a very large market potential in the industrial and electric utility markets throughout the US and the world. Currently GE gas turbines are serving the utility, chemical process, pipeline, and offshore platform markets with a total installed base of 4641 units. Over half of these are used for peaking, mid-range and baseload applications for electric utility power generation and large industrial cogeneration applications. In addition, GE combined cycle systems can benefit substantially from CFCC combustors and shrouds, particularly those utilizing the new advanced gas turbines such as the MS7001F and MS9001F machines.

The advanced technology GE MS7001F and MS9001F sales now total 32 units around the world and market demand is expected to be strong throughout the 90's. Shipments in the range of 15 to 20 units per year (over 3000MW) are likely in the late 1990's.

It is anticipated that trends toward stricter emissions regulation will drive market demand strongly toward upgrade of existing combustion systems for lower emissions. The retrofit market potential is very large; if only 10% of the existing turbines were candidates for the improved CFCC components, this would be about 500 machines by the year 2000.

As CFCC's are introduced on a next generation machine, with no prior constraints as with a retrofit or upgrade application, the advantages, i.e. improved performance and emissions, will be so enormous that their application would contribute significantly to continuation of US leadership in gas turbine technology.

In order to proceed to commercialize the gas turbine CFCC components, a significant development program is required beyond the scope of the proposed DOE CFCC program. At the end of the DOE program Phase III, component feasibility will have been demonstrated from a configuration, materials, and manufacturing processes standpoint. However, the actual design for a full-scale commercial gas turbine needs to be performed and tested. It is anticipated that this would require a significant materials data program to allow component manufacture to begin. While the material supplier is developing the manufacturing process and gathering data, the next step for GE would be to pursue a full-engine test demonstration. An uprate program already defined in the general product plan would be identified for the roll-out of this

technology. The full-engine test demonstration would be structured around the product development schedule. A 7FA uprate would be the ideal model to pursue, given that all the work to date will have been targeted for a 7FA.

A detailed design phase would be initiated for the model uprate targeted. Figure 71 illustrates the commercialization process. Task 2.0, the ongoing methods technology program at universities and labs would directly feed the design phase. Operating requirements will be identified and the system issues and challenges addressed. The results from the design effort will be packaged into detailed specifications for the component and become the basis for manufacturing such parts. The components would be manufactured in the pilot facility created in Phase III. These activities will be followed by full-scale rig testing at GE's test facility to validate performance, analytical design predictions, and component durability. After the full-scale rig testing is successfully completed, there are two options that could be pursued: approach a customer and go directly into planning an on-site field test, or using an aircraft engine as a base, perform an in-house factory test prior to going into the field.

After sufficient period of field testing to solve problems and gain confidence in the durability and performance of the CFCC components, (if an aircraft engine in-house test had already been conducted, the field testing would be less extensive than what is implied here) GE would commit the components to production on a limited basis. Assuming continued field performance success, these components would gradually be phased into other current production models. Concurrently, designs would be put in place for selected field retrofit packages. New designs would begin to incorporate the technology as standard practice.

As this discussion indicates, the incentive to commercialize these CFCC components is very strong. In this industry a 1% performance gain is a major driving force for implementation of the enabling technology; with the shroud and combustion liner a 2% performance gain is potentially achievable. The size of the field retrofit market provides an added incentive relative to the investment of the time and facilities to produce these components.

Figure 71. CFCC gas turbine components path to commercialization.