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CFCC NEWS

A COMMUNICATION OF THE CONTINUOUS FIBER CERAMIC COMPOSITE PROGRAM

Du Pont's SiC/Enhanced SiC Tubes

Big Test Passed

By Phillip A. Craig
Du Pont Lanxide Composites

This spring, 4000-h exposures in a direct-fired coal-burning boiler were completed for Nicalon®/Enhanced SiC tubes manufactured by Du Pont Lanxide Composites. The tubes showed no evidence of corrosion or strength loss.

4000 h Test for Coal Ash Applications Finds No Evidence of Corrosion or Strength Loss in Tubes

A major task that Du Pont Lanxide Composites (DLC) has had within the CFCC Program has been to assess the suitability of CVI SiC/SiC for industrial applications. The primary industrial partner in this work is Foster Wheeler Development Corporation (FWDC), Livingston, New Jersey. FWDC's team led by T.V. Narayanan and DLC's team led by Atul Shah, Jim Weddell, and Bill Patterson developed tasks to test the materials. FWDC's primary interest was the ability of CFCC materials to survive exposure in a coal-fired hot-air heat exchanger. Near the inlet to the heat exchanger, the tubes can become as hot as 2100 to 2300°F (depending on the design) while being exposed to coal ash and flue gas. Near the outlet, the tubes may reach only 1800°F, but the coal ash may contain high levels of alkali condensed out of the flue gas. The level of alkali depends on whether or not the flue gas is scrubbed before entering the heat exchanger.

DLC and FWDC have over the past few years conducted extensive laboratory studies to simulate exposure of standard SiC/SiC and Enhanced SiC/SiC to a hot-air heat exchanger. FWDC conducted two rounds of unstressed exposures of flat specimens to various coal ashes and tem-

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peratures plus synthesized flue gas. ORNL conducted stressed exposures of C-rings. In addition, DLC conducted extensive tensile stressed oxidation testing. These studies indicate that Enhanced SiC/SiC is the material of choice because of its much improved oxidation resistance, especially under externally applied stress.

However, Enhanced SiC/SiC was found to be susceptible to corrosion during exposure to coal ash at 2300°F, and also during exposure to highly alkaline (i.e., due to unscrubbed gas) coal ash at 1800°F. It was determined that Enhanced SiC/SiC should perform well if the maximum temperature is limited to 2100°F and if much of the alkali is scrubbed from the flue gas.

"DLC's CVI composites have good fracture toughness, high specific strength and stiffness through 2200°F with "ceramic grade" Nicalon.

Performance through 2400°F is available with "Hi-Nicalon" and other advanced fibers. The composites are also characterized by low density and excellent thermal shock behavior.

Advantages

The current metal capability in these tests would be defined as "none or low" alkali content at 1800°F. This is a clear example of CFCC material providing advantages in exposure to extreme environments, enabling processes to operate at 300°F hotter temperatures than the current metals can withstand.

The resistance to alkali additions is important to process economics for Foster Wheeler's system design as it quantifies the degree to which flue gas must be scrubbed to remove alkalis. Most systems contain scrubbers now, and these results, tolerating "low" alkali additions, may provide an additional factor of safety or may allow use of a less expensive scrubber.

The CFCC material utilized in this program, Enhanced SiC/SiC, is produced by the CVI process and has high potential for meeting the requirements for high-temperature structural applications. DLC's CVI composites have good fracture toughness, high specific strength and stiffness through 2200°F with "ceramic grade" Nicalon.

Systems Approach

Performance through 2400°F is available with "Hi-Nicalon" and other advanced fibers. The composites are also characterized by low density and excellent thermal shock behavior. Enhanced SiC/SiC is a material variation developed by Du Pont Lanxide Composites made commercially available in January 1993. It provides advanced long life performance through a systems approach to oxidation protection.

"Enhanced SiC/SiC is a material variation developed by Du Pont Lanxide Composites made commercially available in January 1993.

DLC operates several large, commercial-size CVI furnaces with up to 1.4-m-diam capacity for application of BN, C, SiC and other products. "Hi Nicalon" was not utilized in this program in order to keep costs down to meet FWDC's economic goals.

Long-term exposure in a working heat exchanger or boiler is a natural extension of the laboratory studies. FWDC was able to insert a horizontal probe in a direct-fired coal-burning boiler at Gallatin Station, operated by the Tennessee Valley Authority (TVA).

Four short sections of Enhanced SiC/SiC tube (2 in. ID x 3 in. long x 1/8 in. thick) were placed on the probe: two of the specimens were to be exposed for 4000 h and two for 8000 h. No external load was applied during exposure.

It turns out that TVA exposure is much dirtier than the laboratory tests and could expose the specimens to much higher levels of alkali. On the other hand, at the current probe location the temperature generally did not go above 1750°F.

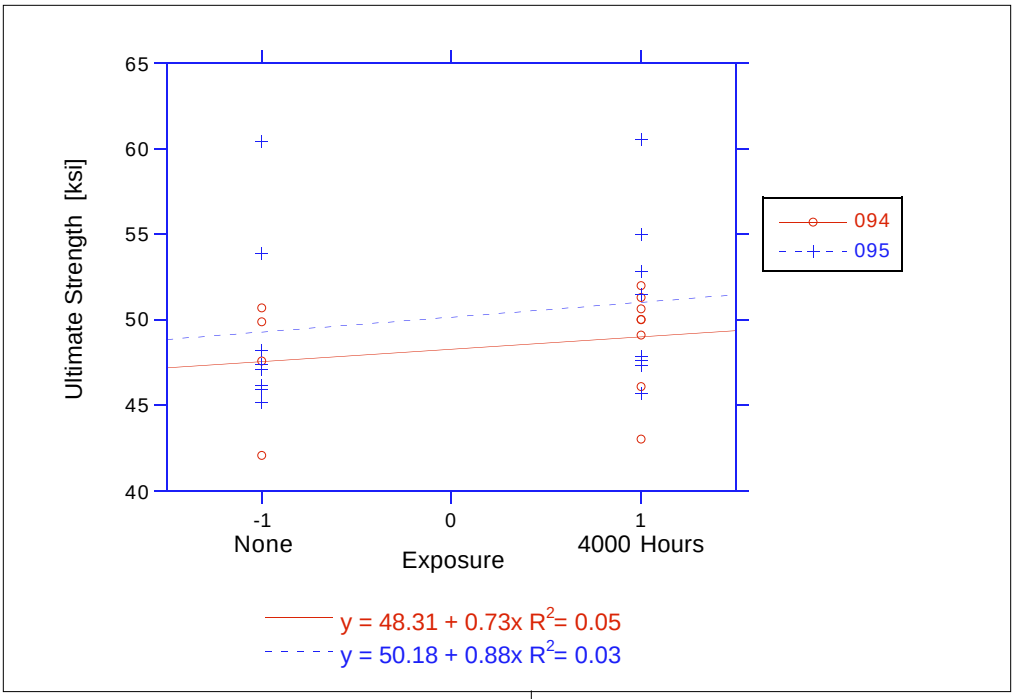

Fig. 1. The linear regressions indicate that exposure had no significant effect on strength. Also, the plot shows good reproducibility between the two tubes. The 8000-h exposures will be completed in August.

No Corrosion, Oxidation

The 4000-h TVA exposures were completed this spring. The specimens had thick layers of coal ash on their outside surfaces, most of which was easily removed. Visual inspection and metallographic examination of the tubes after cleaning showed no evidence of corrosion or significant oxidation in the composite.

There was also no measurable thinning of the tube walls. The tubes were machined into C-rings for residual strength measurement. Both tubes showed no loss of strength due to exposure, see Fig. 1.

Objectives

The objectives of continuing tests in this program are

1. Complete the 8000-h exposures, with follow-up testing and analysis.
2. Perform additional application-specific testing at high temperatures with coal ash exposure and with stress provided by thermal gradient or mechanical load.
3. Perform fabrication and joining work to demonstrate industrial components such as heat exchanger headers and vortex finders, which are exposed to these types of environments.
4. Perform pressure and heat transfer testing of heat exchanger modules. Modules consist of a header and tubes joined together for performance testing at ORNL.▲

Industrial Applications

Fiber-Matrix Control

By Richard Goettler,
McDermott Technology, Inc.

McDermott Technology Oxide-Oxide CFCCs

McDermott Technology, Inc. (MTI) is developing oxide-oxide CFCCs for industrial applications including stationary gas turbine combustor liners, heat exchangers, boiler components and special MTI internal applications.

These CFCC components will require service in high-temperature oxidizing atmospheres for periods of up to three years. A fiber-matrix interface is required in these composites to ensure a damage tolerant material throughout their expected service time.

Oxide-based fiber coatings that provide a weak interface with the fiber and/or matrix and that are nonreactive with the oxide fibers of interest (Nextel™ 610 and Nextel 720™ fiber from 3M Corp.) and the various candidate oxide matrices are desired to meet this need for a long-term damage tolerant ceramic composite.

A fiber-matrix interface is required in these composites to ensure a damage tolerant material throughout their expected service time.

Oxidation Resistance

A significant effort within the MTI CFCC program has been development of oxidation resistant fiber-matrix interfaces for its oxide-oxide composite systems. The typical boron nitride fiber coatings being pursued by many of the nonoxide CFCC developers are not practical for the oxide systems for a number of reasons:

1. A main advantage of the oxide systems is their inherent stability in oxidizing atmospheres and this stability would be jeopardized by introduction of a BN coating.
2. The SiC over coating typically applied to the BN coatings to prevent their oxidation are not stable with the oxide matrices.
3. The porous nature of the oxide matrices resulting from the sol-gel process and the high oxygen diffusivity through oxides would encourage the oxidation of a nonoxide interface.

MTI is pursuing two interface approaches: (1) fugitive interface for use in a MTI internal application and (2) scheelite ($CaWO_4$) for use in other dense structural components.

Fugitive Interface

The fugitive approach are achieved by deposition of a carbon fiber coating, which is present during composite processing and oxidized as the last step of the process to create a fiber-matrix interfacial debond.

The attractive features of this approach are the low-cost precursors available to deposit carbon coatings by solution methods and the inherent debonding achieved at the fiber-matrix interface.

The fiber coatings are deposited using a technique developed by Randy Hay at the U.S. Air Force Wright Laboratory. This laboratory uses hydrocarbons—such as pentadecane—that are immiscible with the coating precursor and serve to reduce bridging between fibers, which often occurs using solution-

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"The attractive features of this approach is the low-cost precursors available to deposit carbon coatings by solution methods and the inherent debonding achieved at the fiber-matrix interface.

based coating techniques.[1] A schematic of the fiber coating apparatus assembled by MTI is shown in Fig. 1.

MTI has coated about 35 lb of Nextel 610™ and Nextel 720™ fiber with carbon. These coated fibers have been woven into 2D and 3D fabrics from which actual components and test specimens have been fabricated. No serious degradation of the carbon coating was observed as a result of the weaving process. Figure 2 shows the stress-strain curve achieved for thin-gauge components (approximately 0.015-in. thick) being supplied to a MTI internal customer. The notable feature is the high strain to failure of 0.28 percent.

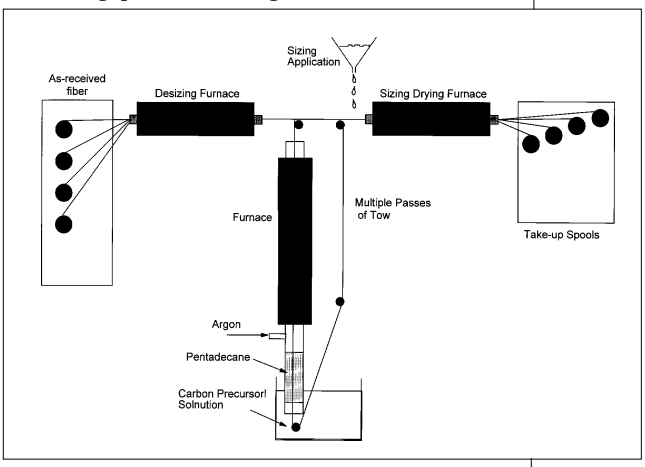

Fig. 1. Schematic of the MTI fiber coater utilizing the immiscible liquid technique.

The fracture surface of the sample, shown in Fig. 3 (next page), reveals interfacial debonding and fiber pull-out. The main issue remaining to be addressed for this interface concept is its stability over time at the anticipated use temperatures.

Exposure of samples for 100 and 200 h in air show minimal strength degradation. Longer exposure times, along with creep rupture data are required to further substantiate this interface approach.

Oxide Fiber Coatings

Oxide fiber coatings are the most desired interfacial solution for oxide-oxide CFCCs because they are inherently stable in the oxidizing use environments being targeted and because they will protect the fibers during processing and component use from strength degrading chemical species. By contrast, the fugitive interface concept leaves the fibers exposed to the processing and use environments.

Initial work in the ceramic community on oxide fiber coatings centered on analogs to the carbon and boron nitride fiber coatings, which are being used extensively by the SiC CFCC community. Layered oxides such as fluorophlogopite, beta-aluminas, and magnetoplumbite have been examined.[2,3,4]

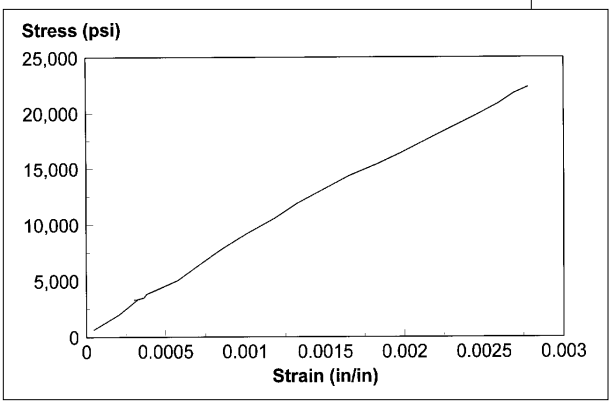

Fig. 2. Stress-strain curve of a Nextel 610™/fugitive interface/ alumina-YAG matrix CFCC.

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"Scheelite was chosen because of its high melting temperature (1580°C) in comparison to other group two tungstates.

The weak-layered crystal planes of these materials, when suitably oriented within a fiber coating, promote crack deflection around the continuous fibers and subsequent frictional fiber sliding. These compounds tend to have chemical stability problems with candidate fibers and matrices, or they tend to require such high processing temperatures to generate the layered crystal structures that fiber strength degradation may occur.

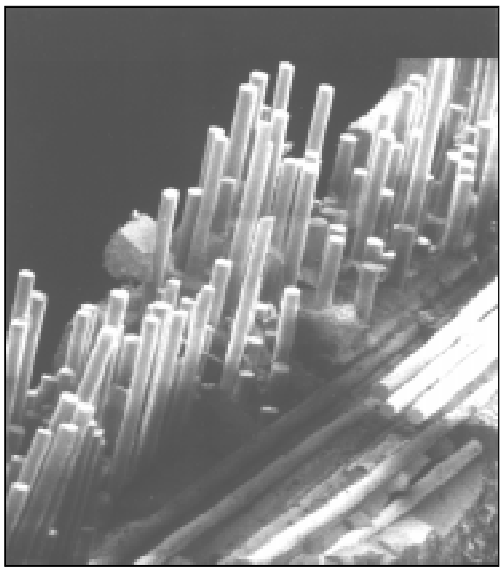

Fig. 3. Fracture surface of the Nextel 610™/ fugitive interface/alumina-YAG matrix CFCC.

Identification of ABO_4 Coatings

Recent work by researchers at Rockwell Science Center has identified two families of nonlayered oxides, monazite ($LaPO_4$) and xenotime (YPO_4), which appear to exhibit non-wetting and weak bonding to fibers containing alumina and alumina silicates.[5]

MTI began an effort to identify additional nonlayered isotropic oxides that would exhibit nonwetting characteristics to oxide fibers. This project is now examining the effectiveness of the coatings in MTI CFCC systems.

These coating compounds would function as effective interfaces in CFCCs by creating high energy interfaces (i.e., being nonwetting) with fibers and/or matrices that would want to readily debond to achieve a lower energy state. Several families of compounds with the general ABO_4 chemical formula including tungstates, molybdates, tantalates, and niobates were identified as promising candidates based on cation valency, cation coordination, and crystal structures (monoclinic and tetragonal phases were preferred for nonepitaxy to hexagonal alumina).

Scheelite Chosen

Initial investigations at MTI have focused on $CaWO_4$ and $ErTaO_4$ coatings. Scheelite was chosen because of its high melting temperature (1580°C) in comparison to other group two tungstates.

The tantalates are of interest because their melting temperature is higher (greater than 1900°C) than the tungstates and because they lack multivalency of the tantalum. The

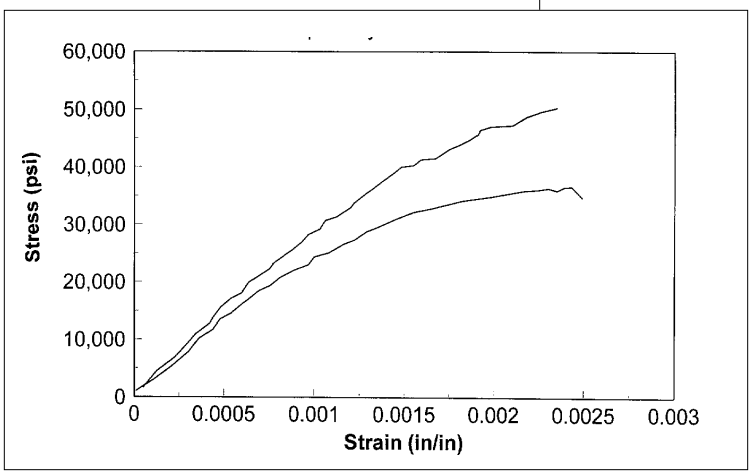

Fig. 4. Stress-strain curve of a Nextel 610™/$CaWO_4$ interface/ alumina-$CaWO_4$ matrix CFCC.

analogous molybdate and niobate compounds are of scientific interest but are probably unsuitable for coatings because of their lower melting temperatures compared to the tungstates and tantalates.

To determine the effectiveness of $CaWO_4$ and $ErTaO_4$ as fiber coatings, unidirectional CFCCs were fabricated using Nextel 610™ fibers. Fiber coatings were applied by using sol-gel precursors developed with the assistance of Sankar Sambasivan at BIRL/ Northwestern University, and the immiscible liquid coating apparatus.

The composites were densified with alumina powder via a pressure filtration technique followed by subsequent infiltration with a concentrated $CaWO_4$ or $ErTaO_4$ precursor. Duplex alumina-scheelite and alumina-$ErTaO_4$ matrices, rather than a pure alumina matrix, were used to avoid possible strength degradation that could occur by use of an alumina polymer infiltrating precursor if the fiber coatings were not uniform.

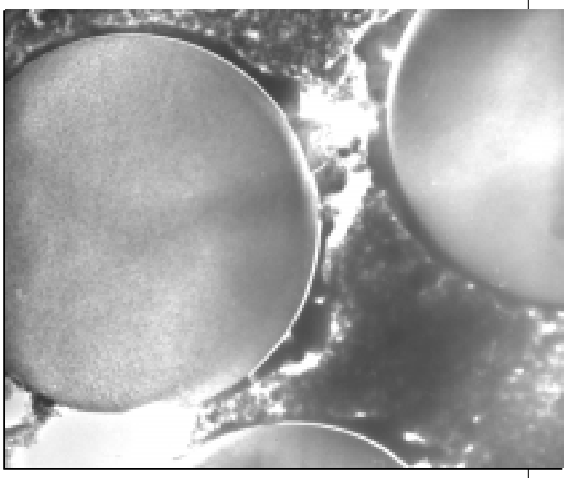

Fig. 5. TEM showing debonding along the scheelite-Nextel 610™ interface.

Composites Densified

The composites were densified to approximately 20% residual porosity. They were exposed to 1100°C for approximately four hours during processing. The $CaWO_4$-containing samples exhibited strains-to-failure of greater than 0.25% (see Fig. 4) and extremely fibrous fracture.

The $ErTaO_4$-containing samples were brittle. Vinayak Dravid of Northwestern University performed transmission electron microscopy (TEM) on these samples to investigate whether any reaction occurred at the interface between the fibers and the fiber coating and to determine if any interfacial debonding is present.

Debonding Observed

Extensive debonding was observed at the scheelite-Nextel 610™ interface supporting the observation of nonbrittle fracture behavior of the specimens (Fig. 5). The $ErTaO_4$-containing specimens did not show any debonding along the interface.

The TEM investigation did not reveal any reaction between either scheelite or $ErTaO_4$ and the fibers. This finding raises the question of why some of the ABO_4 compounds show debonding from fibers (scheelite and monazite) while others do not ($ErTaO_4$), even though all compounds appear stable with the fibers and all have similar crystal structures.

MTI is examining the stability of scheelite with Nextel 720™ fibers. It is expected to be stable based on initial strength retention data obtained for Nextel 720™ fibers coated and heat treated with scheelite. In addition, because scheelite is often found in nature embedded in quartz provides some indirect evidence for likely stability of scheelite and mullite.▲

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Liner Rig Test

Dow Corning and Solar Turbines are Working Together to Develop a Combustor Liner Using SYLRAMIC™ S200 for Land-Based Gas Turbine Engines

By Andy Szweda, Dow Corning, and Vijay Parthasarathy, Solar Turbines

"The use of a CFCC in this application will allow the gas turbine components to be used at higher temperatures, resulting in increased efficiencies, fuel savings, and considerable reduction in emissions.

Dow Corning Corporation has been leading an industrial team effort to develop continuous fiber ceramic composites (CFCCs) for industrial applications. One application being pursued is a combustor liner for land-based gas turbine engines with team member, Solar Turbines.

The use of a CFCC in this application will allow the gas turbine components to be used at higher temperatures, resulting in increased efficiencies, fuel savings, and considerable reduction in emissions.

Initially, a subscale combustor liner has been fabricated for evaluation by Solar Turbines in a hot wall subscale combustor liner rig, where the liner is evaluated for up to 100 h.

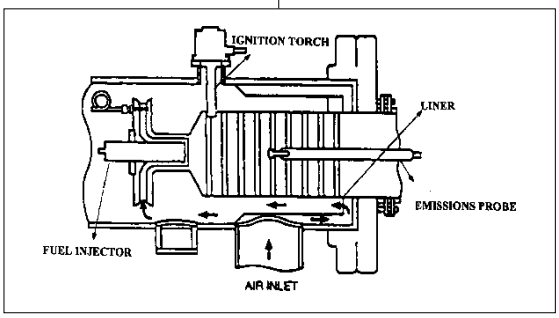

Fig. 1. Schematic of sub-scale combustor rig.

Combustor Rig Test

The subscale rig (Fig. 1) is a high pressure single can rig using a SoLoNox fuel injector. The facility has the capability of achieving the required temperatures and pressures while maintaining a single injector's mass air flow into the rig.

High-pressure air that has been preheated and metered enters the rig near the liners aft end, flows through the fuel injectors, passes through the combustor, and exits the rig through a water quench system. Emissions, temperature, pressure, and flow data are collected during each test.

The purpose of the rig test was to

1. determine thermal gradients along the length and through the thickness of the liner,
2. get a rough order of magnitude estimate on the NO_x and CO emissions, and
3. evaluate the residual mechanical properties after 100 h of rig testing.

CFCCs produced by Dow Corning are fabricated using a polymer impregnation and pyrolysis (PIP) process. This process is a versatile fabrication method that utilizes low-temperature forming and molding steps typically used in organic matrix composites to fabricate large, complex shaped structures.

A SYLRAMIC™ S200 composite was selected for the fabrication of the subscale combustor liner, that consists of a proprietary interface–coated

ceramic grade 8 harness satin NICALON™ SiC fiber in a polymer-derived SiNC matrix. This material has been shown to have good mechanical properties and is recommended for use up to 1200°C.

The architecture selected for the fabrication of the combustor liner was an involute lay-up. This is a architecture where the thickness of the liner is built up using short lengths of plies that are laid up in a overlapping manner on the inside of a cylindrical tool. This architectural approach was taken to facilitate ease of fabrication. A photo of the liner delivered to Solar Turbines is shown in Fig. 2.

Rig Test Results

After approximately 33 h of rig testing of the combustor liner, the rig experienced a dome collapse that caused the rig to be shut down. It was determined through NDE evaluation that this event did not distress the liner. Apart from this one incident, the rig test was uneventful.

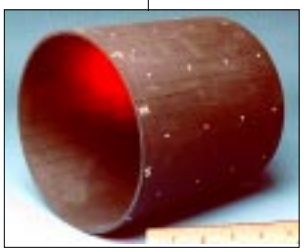

Fig. 2. As-fabricated SYLRAMIC™ S200 combustor liner.

Figure 3 shows a typical variation of wall temperature as a function of time as measured during the rig test. There can be an axial gradient of 1200°F across the length of the combustor. A sudden drop in temperature at certain times indicates that the thermocouple has become detached from the liner. A circumferential gradient can be as high as 400°F in some axial locations.

Emissions

Emissions data obtained from the rig test only roughly simulates the real engine situation but does provide an indication of potential reductions. Emission readings for the SYLRAMIC™ composite liner, corrected to 15% oxygen are in the table below.

	NO_x (ppm)	CO (ppm)
CFCC Combustor	20–40	2–10
Typical Metal Combustor in Engine	50–75	5–15

Post-Test Evaluation

Thermal diffusivity mapping of the subscale liner was carried out by Bill Ellingson at Argonne National Laboratory. NDE was carried out prior to delivery of the liner, after 10 h of testing, after 33 h of testing, and after completion of the 100 h of testing.

No evidence of defects was found in the combustor liner prior to the completion of the 33 h of rig testing, but some features were seen after 100 h of testing. The thermal diffusivity map for the liner after 100 h of rig testing is shown in Fig. 4 and this indicates two regions, located at C and E in the figure, that appear to be evidence of defects.

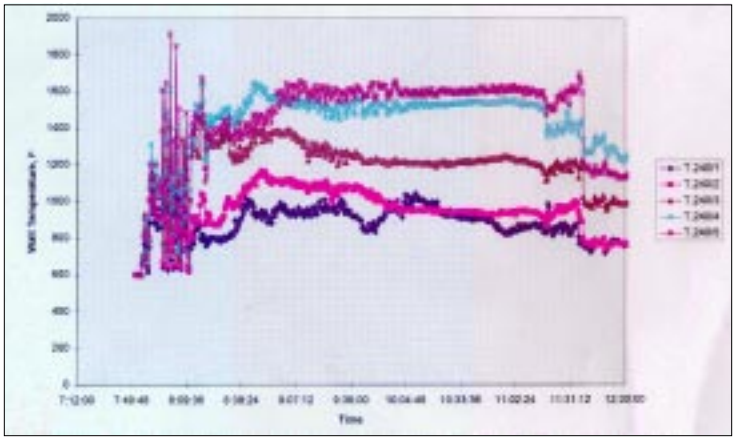

Fig. 3. Typical temperature profile experienced by the combustor liner during a rig test.

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Thermal diffusivity mapping of the subscale liner was carried out by Bill Ellingson at Argonne National Laboratory.

Visual Observations

After the 100 h of rig testing, the liner was visually undamaged (Fig. 5), and no evidence of cracks could be seen. It was evident, however, that the interior surface of the liner had become a light gray color, due to some oxidation, and one particular area on the interior surface was a much lighter color, indicating a greater degree of oxidation (Fig. 6). Microstructural examination indicated that the oxidation was thicker in this particular region. This may indicate that a hot spot was present at this location in the rig. Evidence from the temperature profile for the rig test is still being investigated to support this claim.

The presence of cracks in the liner, as indicated by the NDE, was verified when rings were machined for hoop testing. It was found that two cracks had initiated on the inner (hot) surface. One crack ran axially the whole length of the liner and the other was limited to a 2-in. region. Both of these cracks were on either side of the highly oxidized region.

Another observation was made when sections were cut from the hoop-tested rings that were cut from the liner after the rig test; there was a tendency for the rings to become smaller in diameter. This phenomena was not seen in rings cut from the as-fabricated liner. This is indicative of a stress buildup in the liner during the rig testing.

We are working with Edgar Lara-Curzio of Oak Ridge National Laboratory to determine the magnitude and type of this residual stress, and then determine how this might have occurred. It is possible that the stress was induced by creep of the fiber on the inner hot surface of the liner.

Mechanical Property Testing

Tensile hoop testing and axial flexure testing were carried out on rings cut from the subscale combustor liner before and after the rig test. The data generated is given in Table 1. As can

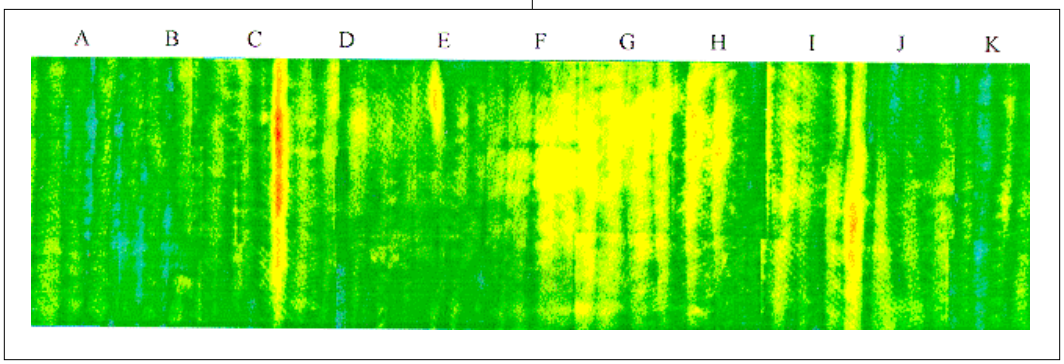

Fig. 4. Thermal diffusivity map of combustor liner after 100 h of rig testing.

Fig. 5. Post-rig-tested SYLRAMIC™ S200 combustor liner.

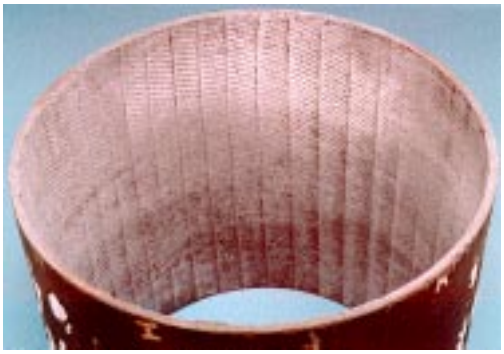

Fig. 6. Interior surface of combustor liner after rig test.

> *"After the 100 h of rig testing, the liner was visually undamaged ,and no evidence of cracks could be seen. It was evident, however, that the interior surface of the liner had become a light gray color, due to some oxidation, and one particular area on the interior surface was a much lighter color, indicating a greater degree of oxidation.*
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> *.*

be seen from these data, there appears to be a significant reduction in hoop strength of the liner after the combustor rig testing.

However, it was determined that each hoop failed at the crack that ran the length of the liner. The axial flexure strength of the material did not change significantly after the rig test, and this may indicate that material degradation did not occur. However, further testing is being carried out by ORNL to verify the data.

In conclusion, the rig test of the SYLRAMIC™ S200 composite liner was a good test. Although the cracks occurred in the liner at some point during the test, the liner did not fail catastrophically, indicating the damage tolerance of CFCCs.

The reason for the cracking is being investigated. The thermal diffusivity mapping NDE technique was shown to be a good method for the detection of cracks in the CFCC liner.▲

	As Fabricated	Post-100 h Rig Test
Hoop Strength (ksi)	13.75	5.22
Axial Flexure Strength (ksi)	34.8	33.9

Table 1. Comparison of as fabricated and post rig test mechanical properties of combustor liner.

”The thermal diffusivity mapping NDE technique was shown to be a good method for the detection of cracks in the CFCC liner.

Successes, Advances Follow from Collaborative Research

Ceramic Joining

By Jill Jonkouski, Department of Energy and R. Bruce Thompson, Ames Laboratory

Recent collaborative research has yielded successful results and advances in the area of joining CFCC’s to themselves and to other materials.

The research is the consequence of the joint efforts of two parts of the Department of Energy (DOE) with distinct purposes: the Division of Materials Sciences (DMS), Office of Basic Energy Sciences (BES), Office of Energy Research (ER), and the Continuous Fiber Ceramic Composite (CFCC) program, Office of Industrial Technologies (OIT), Office of Energy Research and Renewable Energy.

DMS conducts basic research in strategic materials science topics of critical importance to the mission of DOE. The programs are intended to develop the scientific understanding of the synergistic relationship amongst the synthesis, processing, structure, properties, behavior, performance, and other characteristics of materials. Emphasis is placed on the development of the capability to discover new materials and processes.

The CFCC program conducts applied research. The goal is to improve the efficiency and capability of the primary processing methods for the reliable and cost-effective fabrication of CFCC components for use in industrial applications.

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”The goal is to improve the efficiency and capability of the primary processing methods for the reliable and cost-effective fabrication of CFCC components for use in industrial applications.

News Notes

Bill Long Retires: Engineer, Inventor, Editor

Bill Long

Bill Long recently retired from Babcock & Wilcox after 26 years of valued service to the company in the areas of ceramics and advanced materials engineering. For the past several years, Bill provided the company with guidance in program development activities in continuous fiber ceramic composites (CFCC) and in surveying the application of polymer composites for offshore structures in the oil and gas industry.

During his tenure at B&W R&D, Bill had the opportunity to work with most of the company's business units, notably the Insulating Products Division (IPD) in Augusta, Georgia, now Thermal Ceramics since its sale to Morgan in the late 1980s. His work with the staff at IPD led to many significant developments for the company including the coinvention with Helen Moeller of the Rapid-Fire Castable, which was patented and received both the IR-100 and Materials Engineering Awards in 1982. Other work involved fibrous refractory products and Kaowool fiber enhancements. Bill is a holder of four patents in the refractory insulation, fiber, and composites areas. Over the past 10 years Bill has worked closely with the Department of Energy and their Office of Industrial Technology in particular.

He provided valuable industry perspective into the formulation of the very successful DOE Continuous Fiber Ceramic Composite (CFCC) program, and although retired, Bill still assists with program advice through his continuing consultation with B&W. In addition to his contributions at B&W, Bill has been an active participant in SAMPE for many years, serving as editor of the SAMPE Quarterly and having served on their Board of Directors. Bill has also served as B&W's representative to the United States Advanced Ceramic Association (USACA) where he has chaired various working groups.

(From David Baty, McDermott Technology, Inc., formerly Babcock & Wilcox)

CFCCs Noted at DOE's 2nd Industrial Energy Efficiency Symposium

Bill Werst of USACA, right, presented an award during the symposium to Tennessee Congressman Zach Wamp for his contributions and support. Helen Hardin, Wamp's chief of staff, left, received the award for the Congressman.

CFCCs were included in the February 1997 discussions when more than 900 industry representatives met in Arlington, VA, for DOE's 2nd Industrial Energy Efficiency Symposium.

The CFCC program was well represented at the exhibition, with booths on display for Alzeta, Babcock & Wilcox, General Electric, Allied Signal Ceramic Components, Solar Turbines, Dow Corning, Du Pont Lanxide Composites, and Oak Ridge National Laboratory.

Among the highlights of the gathering:

- CFCCs were discussed during one of the plenary sessions at which Allen Womack, senior vice president and chief technical officer, Babcock & Wilcox Power Generation Group, discussed advanced materials research and development.
- Awards for outstanding contributions went to John Oleson, director, manufacturing technology, Dow Corning, and Bill Long, who recently retired from Babcock & Wilcox.

Continued on page 15

Time-Dependent CFCC Deformations Studied

Oak Ridge National Laboratory & Dow Corning

By Edgar Lara-Curzio,
Oak Ridge National Lab, and
Ronald P. Boisvert,
Dow Corning Corporation

During the past three years, the Dow Corning Corporation and Oak Ridge National Laboratory (ORNL) have been involved in a cooperative research and development agreement (CRADA) to investigate the mechanisms responsible for the time-dependent deformation of CFCCs at elevated temperatures.

Recently, Karren L. More of ORNL joined this effort, and the CRADA was extended for an additional 18 months to investigate environmental effects on the mechanical performance of these materials at both intermediate and elevated temperatures.

The materials used in this investigation are being studied by Dow Corning as part of their participation in the DOE-CFCC Program. These materials consist of continuous fibers coated using a proprietary composition, embedded in a polymer-derived amorphous SiNCO matrix.

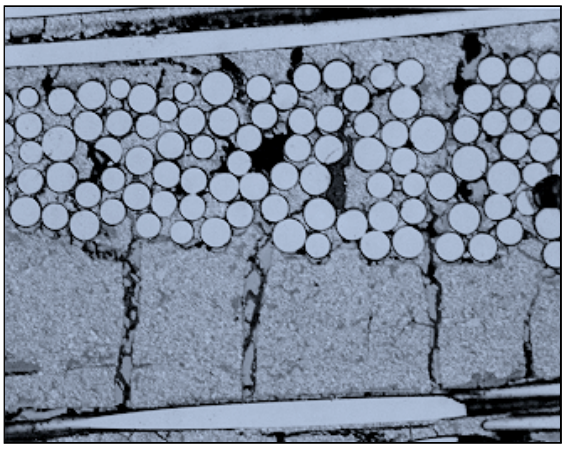


Fig. 1. Optical micrograph of polished section of CG-Nicalon™/SiNCO CFCC.

The composite is densified by the polymer-impregnation and pyrolysis (PIP) method.[1] The micrograph in Fig. 1 illustrates the complex microstructure of the composite. Visible in the micrograph are longitudinal and transverse CG-Nicalon™ fibers with diameters ranging between 12 and 18 µm, the SiNCO matrix, and matrix cracks that result from the multiple matrix infiltration and pyrolysis cycles necessary to achieve densification.

As part of the characterization of the material, a number of mechanical tests have been conducted at temperatures between 600°C and 1200°C. Materials with different fiber architectures (unidirectional, 0/90 8-harness satin weave, 0/90 5-harness satin weave, and ±45° layups of unidirectional plies) and different fibers (CG-Nicalon™, Hi-Nicalon™, and Sylramic™ SiC fiber) have been and will be evaluated.

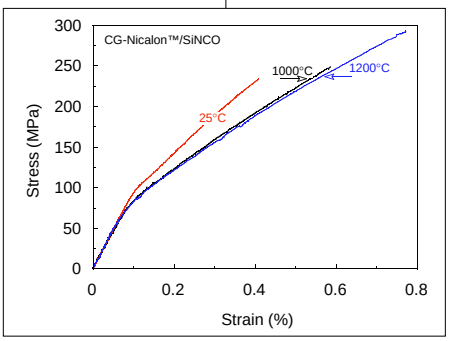


Fig. 2. Monotonic tensile stress-strain curves of CG-Nicalon™/SiNCO CFCC obtained at three different temperatures in air.

The mechanical tests that have been

Supporting Technologies

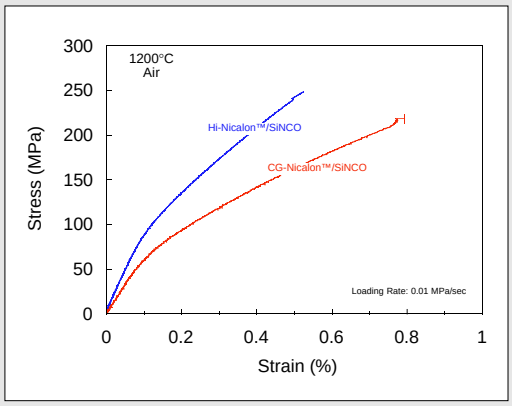


Fig. 3. Monotonic tensile stress-strain curves of CG-Nicalon™ and Hi-Nicalon™-reinforced SiNCO.

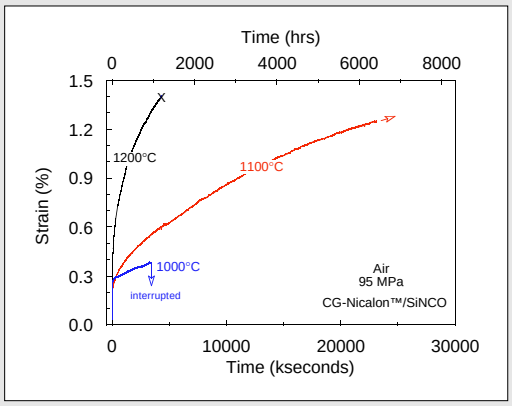


Fig. 4. Strain histories recorded during the creep evaluation of CG-Nicalon™/SiNCO CFCCs at 95 MPa and three different temperatures.

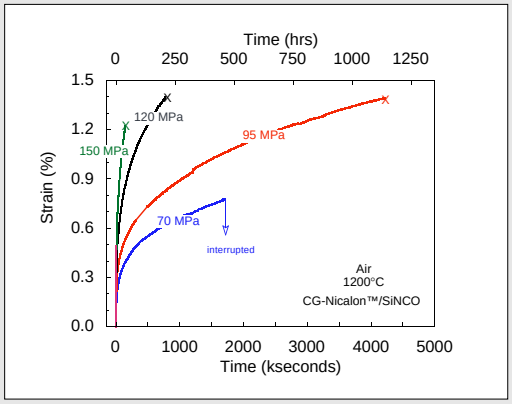


Fig. 5. Strain histories recorded during the creep evaluation of CG-Nicalon™/SiNCO CFCCs at 1200°C under different stresses.

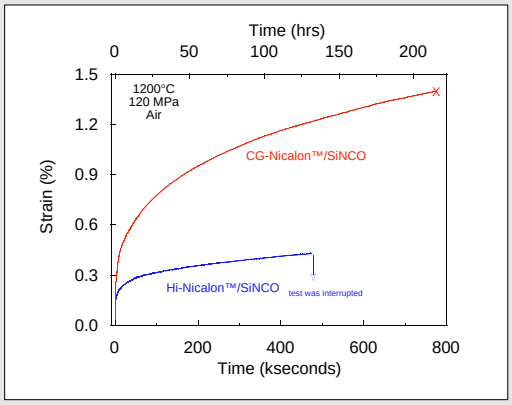


Fig. 6. Strain histories recorded during the creep evaluation of CG-Nicalon™ and Hi-Nicalon™–reinforced SiNCO at 1200°C and 120 MPa.

conducted include monotonic tensile loading at different loading rates, creep, creep recovery, stress-rupture, and stress-relaxation.

Figure 2 shows the stress-strain response of CG-Nicalon™/SiNCO under monotonic tensile loading at three different temperatures. The stress-strain curves are characterized by a linear region up to the so-called proportional stress limit.

This value of the stress (≈ 75 MPa) at the onset of nonlinear stress-strain behavior correlates well with the onset of significant acoustic emissions.[2] At stresses larger than the proportional stress limit, the material exhibits nonlinear stress-strain behavior up to failure.

Temperature Effect

The effect of temperature on the stiffness of the CFCC is apparent in Fig. 2, as well as the increased strain at failure with increasing temperature, resulting from creep deformation. Similar tests have been conducted with composites containing different fibers.

Figure 3 shows a comparison of the tensile stress-strain response of CG-Nicalon™ and Hi-Nicalon™ -reinforced SiNCO CFCCs. The effect of higher stiffness of Hi-Nicalon™ fibers (≈ 350 GPa) over CG-Nicalon™ fibers (≈ 200 GPa) on the stiffness of the CFCC is evident in Fig. 3.

Also evident is the higher strength retention of Hi-Nicalon™ fibers after processing. The curves in Fig. 3 were obtained during monotonic tensile tests conducted at 1200°C at a loading rate of 0.01 MPa/s.

Viscoelastic

It has been found that the time-dependent behavior of these materials at elevated temperatures can be described as viscoelastic in nature. Figure 4 shows the strain histories recorded during the creep evaluation of CG-Nicalon™/SiNCO at 1000°C, 1100°C, and 1200°C and at a constant tensile stress of 95 MPa. It has been

found that the life of CG-Nicalon™/SiNCO composites exceeds 7000 h at 1100°C and 95 MPa. Figure 5 shows the effect of stress on the time-dependent deformation of CG-Nicalon™/SiNCO at 1200°C.

It should be pointed out that the changes in compliance recorded during creep tests, and labeled as "strain" in Figs. 4 and 5, result from the intrinsic flow of the composite constituents at elevated temperatures (creep), from changes in compliance resulting from both fiber and matrix failure, and from "stretching" of the fiber weave structure.

The recent availability of stoichiometric SiC fibers has expanded the window of operations of CFCCs to higher temperatures. In contrast to CG-Nicalon™ fibers, fibers such as Hi-Nicalon™ and Sylramic™ offer improved creep resistance and environmental stability.

Creep Resistance

The higher creep resistance of Hi-Nicalon™ fibers over CG-Nicalon™ fibers is well evidenced in Fig. 6. The figure shows the strain histories of tests conducted at 1200°C and a tensile stress of 120 MPa on both CG-Nicalon™ and Hi-Nicalon™-reinforced SiNCO. Note that after 100 h, there is a factor of five difference in the magnitude of the rate of deformation between these materials.

The information being collected in this program will be very valuable for design purposes, and for understanding the intrinsic behavior of the composite constituents, and fiber architecture, on the time-dependent behavior of CFCCs at elevated temperatures.▲

References

1. A. Szweda, R. Boisvert, and R. Jones, "Materials/Processing Methods for Continuous Fiber Ceramic Composites," pp. 1791–1805 in *Proceedings of the 39th International SAMPE Symposium*, Anaheim, California, April 11–14, 1994, .

2. E. Lara-Curzio, M. Ferber, R. Boisvert, and A. Szweda, "The High Temperature Tensile Fatigue Behavior of Polymer-derived Ceramic Composites," *Ceram. Eng. and Sci.*, **16** (4), 341–349 (1995).

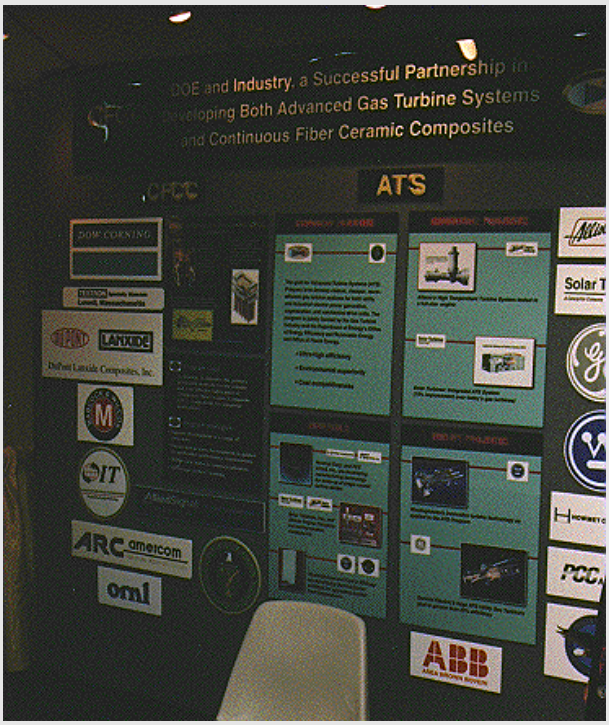
News Notes

— Continued from page 13

■ *CFCC News* Wins Publications Award

CFCC News, a communication of the CFCC Program, was given the 1996 Achievement in Newsletters award by the East Tennessee Chapter of the Society for Technical Communication (STC). This is the second STC award won by *CFCC News* in the technical publications competition.

Gloria Caton is the managing editor, and Tim Elledge is the writer, editor, and designer of the publication. The winning newsletter was on display at the American Museum of Science and Energy in Oak Ridge.



Part of the joint CFCC–ATS display at DOE's 2nd Industrial Energy Efficiency Symposium held in February 1997 in Arlington, Virginia.

Ceramic Joining

—— Continued from page 11

The BES and CFCC researchers discuss the results of their work and plans for the future in the open "BES/CFCC Joining Workshops," to which all interested parties are invited to attend. The workshops are normally held in conjunction with large technical meetings such as the Annual CFCC Program Review and the Annual Conference on Composites, Materials, and Structures (Cocoa Beach, Florida).

The successes and advances resulting from the extremely fruitful collaborations, which started in April, 1995, are detailed in the paragraphs that follow.

New Joining Technology

The work described here is funded jointly by CFCC and BES. A short history and current status of the work are included in each description, followed by contact information.

• Ames Lab has joined Dow Corning's CFCC using a metal-filled pre-ceramic polymer at 1200°C in air. Flexture testing of the butt-joined samples yielded strengths of 34 MPa at both room temperature and 600°C. Dow Corning's SEM analysis identified embedded alumina particles in the joint. Following this successful experiment, a new set of CFCC samples has been sent to Ames Lab to be joined using the same approach, with different pre-ceramic polymers. The joint will be used in the sealless chemical pump-shell application.

Contacts:
Beebhas Mutsuddy, Dow Corning
Sina Maghsoodi, Ames Lab
Özer Ünal, Ames Lab

• Ames Lab has received CFCC material from Du Pont Lanxide Composites (DLC). Both butt and lap joints, joining CFCC to CFCC, have been fabricated using a metal-filled pre-ceramic polymer and DLC's Ceraset™ pre-ceramic polymer. The CFCC butt joints were tested using a 4-point bend test. Up to 800°C there has been only a 20% loss of strength, compared to 80% when the work first started. Testing continues at room temperature and at high temperatures. The next step is to join a geometric shape representative of the intended application.

Contacts:
Sina Ijadi-Maghsoodi, Ames Lab
Phil Craig, Du Pont Lanxide
Özer Ünal, Ames Lab
Iver Anderson, Ames Lab

• Ames Lab has received CFCC material from Atlantic Research Corporation to be joined in a CFCC-to-CFCC joint. The CFCC was reinforced with Nicalon CG and high-Nicalon. Butt joints were fabricated and tested using a 4-point bend technique. Reasonable strengths were obtained. The next step is to join component-sized pieces for the actual OEM application.

Contacts:
Jim Bird, Atlantic Research
Sina Ijadi-Maghsoodi, Ames Lab
Özer Ünal, Ames Lab
Iver Anderson, Ames Lab

• Battelle Pacific NW National Laboratory has received Dow Corning CFCC samples for CFCC to-metal bonding using a transition metal bonding approach. The joints will be fabricated during the1997 summer season.

Contacts:
Beebhas Mutsuddy, Dow Corning
Chuck Henager, PNNL

• Oak Ridge National Laboratory (ORNL) evaluated the compatibility of two active brazing filler materials on Dow Corning's CFCC, Sylramic 100. Two commercial alloys were used, each of which solidified at different temperatures. Metallographic examination showed that both filler metals produced joints that were well filled with no signs of delamination of the CFCC, excessive porosity, or unbonded regions.

One of the brazes produced thin, continuous reaction layers. Also, several experiments were done to evaluate the brazing of the CFCC to monolithic silicon carbide and silicon nitride.

Contacts:
Mike Santella, ORNL
Beebhas Mutsuddy, Dow Corning

• Dow Corning and ORNL performed experiments to evaluate the wetting behavior of a borosilicate glass on Sylramic 100 for use in a potential joint.

Contacts:
Beebhas Mutsuddy, Dow Corning
Mike Santella, ORNL

• CFCC samples have been sent to Lawrence Berkeley National Laboratory (LBL) to be joined using a transient liquid phase bonding approach.

Contacts:
Andy Glaeser, LBL
Phil Craig, Du Pont Lanxide

Finite Element Modeling (FEM)

• Dow Corning provided details of the sealless pump component to

Idaho National Engineering & Environmental Laboratory (INEEL), including dimensions, drawings, and environmental conditions. INEEL performed a FEM analysis. INEEL concluded that direct diffusion bonding was not going to be possible. Modeling with a braze alloy showed very large stresses, possibly too large. Dow Corning is presently designing a joint based on INEEL's recommendations and the customer's input (Sunstrand Fluid Handling). The modeling suggested changes in geometry that could be used right away—such as rounding of corners and making the structure very compliant. One possible solution is a graded joint.

Contacts:
Beebhas Mutsuddy, Dow Corning
Barry Rabin, INEEL

Related Joining Work

• Du Pont Lanxide Composites Inc. (DLC) and INEEL have been working together on reaction-bonded joining techniques for the combustor liner application and the heat exchanger applications. They have demonstrated joint strengths equal to that of the DLC CFCC composite, using double-notch shear testing at room temperature. Future activities include looking at high temperature properties (fracture toughness), and joining tubular components.

Contacts:
Phil Craig, Du Pont Lanxide
Barry Rabin, INEEL

• Sandia National Laboratories will commence work with GE, Du Pont Lanxide Composites, Babcock & Wilcox, and Dow Corning. CFCCs will be joined using oxide-based glasses. (Past work with these materials joining Si_3N_4 was very successful. The average strength of the oxynitride glass joints was 550 MPa at 1000°C, using 4-point bend testing, higher than any previously reported for this temperature.)

The oxide-based glass formulations are favorable for the non-oxide ceramics because they are chemically stable, have high softening points, and can be crystallized to even more refractory phases that also will allow control of thermal expansion.

• Sandia National Laboratories and Amercom will commence work, during the 1997 summer season, on a braze technique to be used for ceramic-to-metal joining. The braze will function at high temperatures and has the capacity to relieve thermal expansion mismatch stresses generated between dissimilar materials.

Contacts:
S. Jill Glass, Ron Loehman, SNL
Bill Darden, Textron
Phil Craig, Du Pont Lanxide
Beebhas Mutsuddy, Dow Corning
Krishnan Luthra, General Electric

Adjacent Activities

A number of related collaborations have been initiated as the result of tangential interest to the BES/CFCC joining work and contacts made in the workshops.

• Through the CFCC Task 2 program, using the special one-on-one activity area funding, Argonne National Laboratory (ANL) and Dow Corning are assessing NDE methods that will be used to detect variations in processing conditions. As a part of this assessment, Dow Corning sent three lots of joined PIP-CFCC samples to ANL for NDE.

Four different processing conditions will be evaluated via thermal diffusivity, air-coupled ultrasound, acoustic resonance, and laser holographic interferometry. Prior NDE data, using thermal imaging methods, have suggested the ability to determine the extent of infiltration of a pre-ceramic polymer in a joint.

Contact:
Bill Ellingson, ANL
Beebhas Mutsuddy, Dow Corning

• A four-way cooperation between INEEL, ANL, Stone & Webster Engineering Corp., and ORNL is underway. INEEL joined a monolithic SiC tube having a 3-in. diameter and a 0.25-in. wall thickness. ANL performed an NDE evaluation, including X-ray computed tomographic imaging for density variations around the joint, and acoustic resonance.

The results from each technique agreed with the other. In addition the results agreed with the theory of acoustic resonance of solid-walled tubes and vibratory modes (discontinuous wall around solid-walled tube, that is, an incomplete joint).

The joint will be exposed at ORNL in a special steam exposure furnace, and the joint will be returned to ANL for more NDE. INEEL is joining a 0.5-in.-diam, 7 ft long tube, at present, for SWEC. This work is funded by OIT's Advanced Heat Exchanger Program and BES.

Contacts:
Barry Rabin, INEEL
Bill Ellingson, ANL
Joe Gondolfe, SWEC
Jim Keiser, ORNL

Conclusion

This collaborative research has had two major consequences. First, from the perspective of basic research, the experiences gained by the BES researchers in applying their ideas to the CFCC problems have identified numerous new directions of important scientific studies.

Second, from the perspective of technology application, the techniques being developed in these BES/CFCC collaborations are reducing the effect of joining of dissimilar materials as a rate-

limiting factor of the CFCC program.

Many advances have been made. Problems are being overcome with candidate solutions, and test data are being generated on the new joints. The joints must yet be demonstrated on real parts in the field.

The "BES/CFCC Joining Workshops" provide informal settings for idea exchange and encouragement. Proceedings of the three workshops, which have been held to date, are available by contacting R. Bruce Thompson at Ames Laboratory.

Several papers on the joining research discussed here were presented at the American Ceramic Society Annual Meeting. Copies of the proceedings can be obtained by contacting Iver Reinanis, Colorado School of Mines.

Joining Investigators, Research Areas

Iver Anderson, Ames Lab
Phone: 515-294-8252, FAX: 515-294-8727
E-mail: andersoni@ameslab.gov

- *Joining techniques for SiC based on toughening preceramic polymers with metal particles.*

James O. Bird, Atlantic Research Corp.
Phone: 508-658-8940, FAX: 508-658-2306
E-mail: jbird@arc-ag.com

- *Develop processing methods to produce reliable and cost effective CFCC components for industrial applications (hot gas circulation fans, hot gas filters, diesel engine piston rings) employing a chemical vapor infiltration process for densification.*

Phillip A. Craig, Du Pont Lanxide Composites Inc.
Phone: 302-456-6577, FAX: 302-456-6480
E-mail:craigpa@cdcux05.lvs.dupont.com

- *Develop processing methods to produce reliable and cost effective CFCC components for industrial applications (heat exchanger tubes, combustor liner, tip shrouds) using the DIMOX™-directed metal oxidation process.*

Bill Darden, Textron Systems Division
Phone: 508-452-8961 ext. 5224, FAX: 508-454-5441
E-mail: wdarden@tsd.textron.com

- *Develop processing methods to produce reliable and cost effective CFCC components for industrial applications (immersion tube burner, combustor liner) using a slurry-infiltration process that when fired, produces a nitride-bonded silicon carbide matrix.*

William A. Ellingson, Argonne National Lab
Phone: 630-252-5068, FAX: 630-252-4798
E-mail: wa_ellingson@qmgate.anl.gov

- *Develop nondestructive characterization methods for characterizing as produced, repaired, and used joints. These will be CFCC as well as monolithic lap joints, butt-joints, and t-joints.*

Andy Glaeser, Lawrence Berkeley National Lab
Phone: 510-486-7262, FAX: 510-486-6086
E-mail: aglaeser@sapphire.berkeley.edu

- *Design of novel, low-temperature approaches to elevated-temperature, ceramic-ceramic and ceramic-metal joints utilizing transient liquid phases.*
- *Processing, microstructure, property relations for joined materials.*

S. Jill Glass, Sandia National Laboratories
Phone: 505-845-8050, FAX: 505-844-4816
E-mail: sjglass@sandia.gov

- *Use of oxide glasses to join silicon nitride and silicon carbide.*
- *Brazing.*

Joe Gondolfe, Stone & Webster Engineering Corporation
Phone: 281-368-4379, FAX: 281-368-3500
E-mail: joseph.gondolfe@stoneweb.com

- *Develop an advanced ceramic furnace for ethylene production (expected to increase ethylene production by up to 10%).*

Paul Gray, Du Pont Lanxide Composites Inc.
Phone: 302-456-6376, FAX: 302-456-6480
E-mail: graype@cdcux05.lvs.dupont.com

- *Develop processing methods to produce reliable and cost-effective CFCC components for industrial applications (combustor liner, tip shroud, heat exchanger tube, porous radiant surface burners, reverberatory screens) using a chemical vapor infiltration process for densification.*

Chuck Henager, Pacific Northwest National Lab
Phone: 509-376-4276, FAX: 509-376-0418
E-mail: ch_henager@pnl.gov

- *High-temperature mechanical properties of ceramic composites.*
- *Displacement reaction joining methods.*

Krishnan Luthra, General Electric R&D
Phone: 518-387-6348, FAX: 518-387-7007
E-mail: luthra@crd.ge.com

- *Develop processing methods to produce reliable and cost-effective CFCC components for industrial applications (combustor liner, shroud) using Toughened Silcomp™ ceramic composite materials.*

Sina Ijadi-Maghsoodi, Ames Lab
Phone: 515-294-1110, FAX: 515-294-4456
E-mail: ijadi@ameslab.gov

- *Use of preceramic polymer-based materials to join silicon carbide/silicon nitride ceramics.*
- *Processing, microstructure evaluation.*

Jill Jonkouski, U. S. Department of Energy, Chicago
Phone: 630-252-2657, FAX: 630-252-8649
E-mail: jill.jonkouski@ch.doe.gov

- *Project Manager (CFCC).*
- *BES/CFCC "Joining Workshop" co-chair.*

Jim Keiser, Oak Ridge National Lab
Phone: 423-574-4453, FAX: 423-574-5118
E-mail: keiserjr@ornl.gov

- *Testing of joined coupons and tubes at high temperature and in steam and other environments.*

Ron Loehman, Sandia National Lab
Phone: 505-272-7601, FAX: 505-272-7304
E-mail: loehman@sandia.gov

- *Use of oxide glasses to join silicon nitride.*
- *Brazing.*

Beebhas Mutsuddy, Dow Corning Corporation
Phone: 517-496-6904, FAX: 517-496-6278
E-mail: usdccqvj@ibmmail.com

- *Develop processing methods to produce reliable and cost-effective CFCC components for industrial applications (combustor liner, interstage seal ring, pipe hangers, containment shell) using the polymer impregnation and pyroysis process.*
- *Evaluate different methods of joining CFCCs to similar and dissimilar materials.*

Barry H. Rabin, Idaho National Engineering Lab
Phone: 208-526-0058, FAX: 208-526-0690
E-mail: bhr@inel.gov

- *Studies of residual stresses and thermo-mechanical behavior of dissimilar material joints.*
- *Ceramic joining via reaction bonding.*

Iver Reimanis, Colorado School of Mines
Phone: 303-273-3549, FAX: 303-273-3795
E-mail: ireimani@mines.colorado.edu

- *Fracture and deformation of ceramic/metal joints with graded microstructure.*

Mike Santella, Oak Ridge National Laboratory
Phone: 423-574-4805, FAX: 423-574-4928
E-mail: santellaml@ornl.gov

- *Advanced brazing techniques.*

R. Bruce Thompson, Ames Laboratory
Phone: 515-294-9649, FAX: 515-294-4456
E-mail: thompsonrb@ameslab.gov

- *Project Coordinator (BES Materials Joining).*
- *BES/CFCC "Joining Workshop" co-chair.*

Özer Ünal, Ames Laboratory
Phone: 515-294-4892, FAX: 515-294-8727
E-mail: unal@ameslab.gov

- *High temperature properties, fracture, fatigue.*

Task 2 Reviewed

The Firing Line

By Peter Tortorelli,
Oak Ridge National Laboratory

> *"The feedback from the review panel was extensive and varied.*

> *"In the Task 2 response, most of the points regarding approaches, redirection, issues, research areas, etc. were addressed to some degree.*

Within the past six months, the CFCC Supporting Technologies Program (Task 2) has been reviewed in a process that included the following:

1. Presentations by Task 2 personnel to a review panel on December 18, 1996, in Washington, D. C. The panel consisted of representatives of various DOE Offices (Industrial Technology, Transportation Technology, Energy Research, Chicago Operations), and of companies/individuals involved with the Task 3 efforts.
2. Collection of comments from individual members of the review panel by DOE and transmittal of these (in a nonattributed format) to the Task 2 managers and principal investigators (PIs).
3. The formulation of the Task 2 response, including planned actions, which was forwarded to DOE in late March and followed by a briefing to the CFCC Program Steering Committee on April 2, 1997, by Michael Karnitz.

The morning part of the December 18 meeting involved summaries of the various components of Task 2. Topics and presenters were Task 2 Introduction, Michael Karnitz; Composite Design and Interfaces, Richard Lowden; Materials Characterization, Karren More; Test Methods, Edgar Lara-Curzio; Performance-Related Phenomena, Peter Tortorelli; Modeling, Edgar Lara-Curzio; Nondestructive Characterization, William Ellingson.

In the afternoon, two CFCC-related ORNL CRADAs were reviewed for the DOE members of the review panel. The others met in a parallel session for initial discussions regarding the review process and the nature of the resulting comments and recommendations.

> *Fourteen different areas were identified for specific responses by Task 2.*

The feedback from the review panel was extensive and varied. It included quite favorable comments as well as complaints, practical suggestions, philosophical questions, and recommendations and critiques that ranged from very specific to quite general.

Response

In the Task 2 response, most of the points regarding approaches, redirection, issues, research areas, etc., were addressed to some degree. Given the sheer volume of the feedback, comments requiring a response were clustered when they represented the same or a related issue to eliminate redundancy, highlight common themes, and limit the document to a reasonable size.

Fourteen different areas were identified for specific responses by Task 2:

1. Roles of the Supporting Technologies Task.

2. Industrial liaisons/relevance of work.
3. Establishment and development of databases/standard test procedures.
4. Design specifications and methodologies.
5. Suggested research areas.
6. Fiber coatings/interfaces.
7. Life prediction.
8. Nondestructive characterization.
9. Materials Sciences Corporation modeling.
10. ORNL modeling.
11. Performance-Related Phenomena subtask.
12. Interaction with the Enabling Propulsion Materials Program.
13. CFCC hardware/combustor liners.
14. Benchmarking.

Space limitations preclude inclusion of the fourteen sets of comments/recommendations and the respective responses. However, a brief summary of the key concerns, some general response comments, and a list of follow-on actions follow.

Messages

After evaluation of the numerous comments from the review panel, several dominant messages emerged. These included

- assure industrial relevance,
- be more inclusive with respect to the industrial participants,
- increase emphasis on environmental effects and alternative interfaces, and
- continue efforts to promote standards development and help establish databases.

Industrial Interactions/Materials

In the limited time allotted for the December review meeting, it was not possible to cover all the activities in Task 2. This may have led to some concerns regarding the number and nature of industrial interactions and the types of materials being examined.

Perusal of the feedback indicated that a number of the reviewers perhaps felt that Task 2 studies have focused too much effort on CFCCs with carbon interface coatings or composites produced at ORNL.

This was the obvious starting point as, at the beginning of the CFCC Program, such materials were the most highly developed and most readily obtainable. Nonetheless, a wide variety of non-oxide based, and some oxide-based, composites have now been tested, evaluated, and characterized, and these results have been included in the bimonthly reports when the information was not held in confidence (see below).

Some of the variety of CFCCs characterized and evaluated in Task 2 is indicated by the list in Table 1 (page 23). A principal reason for the range of different composites has been the relationships established with several of the CFCC industrial participants, which have led to obtaining materials to supplement what Task 2 has been able to procure using its own funds or other avenues. This is important as the need for characterizing the most current developmental materials is critical to the success of the Program. As shown by some parts of the December 18 review, Task 2 efforts in recent years have increasingly involved successful interactions with several of the industrial participants.

These efforts were facilitated by the informal use of designated Task 2 contacts and, in response to reviewer input, Task 2 industrial liaisons have now been named for each of the Task 3 contractors. It is felt that these liaisons will not only help Task 2 in its effort to investigate relevant materials, but help address the panel recommendations associated with all four of the major areas listed above.

A key issue in establishing working relationships with the Task 3 partici-

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A key issue in establishing working relationships with the Task 3 participants is that of the need to keep data confidential until cleared by the company who provided the particular material.

company that provided the particular material. When the material has been supplied at no cost to Task 2, the data is shared with the provider and not released to the public domain until cleared by the industrial partner.

It is believed that, generally, Task 2 has been satisfactorily dealing with this confidentiality issue over the past several years, even when it leads to the perception (on the part of several reviewers and others) that the Supporting Technologies efforts are focused on "old" or irrelevant composites.

Role of Task 2

Some of the individual comments included in the input from the review panel reflected some misunderstanding of the role of Task 2 in the CFCC Program. Consistent with its charter, the Task 2 efforts provide generic and precompetitive R&D and characterization as guided by the needs of Task 3 projects and the targeted applications for CFCCs.

To this end, a principal objective of Task 2 is to provide important insights into the factors (e.g., stability of fibers, interfacial layers, matrix cracking) controlling the service life and reliability of CFCCs over a wide range of temperatures and environmental conditions. The emphasis of Task 2 has shifted to more industrially relevant efforts as Task 3-produced materials have become available and CFCC applications have been better defined.

Evaluation of the effects of various gaseous environments were initiated using test facilities that have been developed with input from the industrial participants. This latter effort was undertaken to specifically address the service conditions that were expected.

Unfortunately, every possible system environment cannot be simulated or investigated; the Task 2 approach is to address the critical gaseous environmental constituents (e.g., partial pressure of oxygen, water vapor, etc.) based on the constituent phases in the CFCCs being developed, as well as testing composites that might provide enhanced performance. Task 2 PIs have specifically sought to identify the constituent phases and reactions that control the life and reliability of each CFCC for a range of test conditions. In so doing, it is believed Task 2 is making the best use of its collective expertise and resources consistent with its charter.

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Follow-up

Several actions have been or will be taken as a result of the Task 2 review. As already mentioned, liaisons for all Task 3 teams have now been named.

Other examples include an expansion of composite systems, with a focus on key issues using well-defined environmental and material experimental approaches, and a greater emphasis on relating modeling and life prediction to industrial needs.

In the latter area, specific actions include better coordination of these efforts by having Edgar Lara-Curzio of ORNL take responsibility for such, more extensive use of modeling and life prediction as applied to real CFCC components and applications, and a recommendation for a Task 2-sponsored session on these approaches at the next CFCC Working Group meeting.

The Task 2 managers and PIs found that the December 18, 1996 meeting, the associated feedback, and the effort to prepare the responses were valuable in assessing progress, communicating with Task 3 participants and other people interested in CFCC-type materials, and planning future work.

It appears that DOE and other program participants also felt the review process was worthwhile. It is anticipated that such a review will now occur on an annual basis.

Matrix	Reinforcement	Interfacial layer	Seal Coat	Manufacturer
FCVI SiC	Nicalon plain weave	C	With and w/o CVD SiC	ORNL
FCVI SiC	Nicalon plain weave	B-Doped C		ORNL
FCVI SiC	Nicalon plain weave	BN		ORNL
CVI SiC	Nicalon plain weave	C	With and w/o CVD SiC	B. F. Goodrich
CVI SiC	Nicalon plain weave	C	With and w/o CVD SiC	DuPont
FCVI SiC	Nextel 312 plain weave	C		ORNL
FCVI SiC	Nextel 312 & 440 plain weave	BN		ORNL
FCVI SiC	Nextel 440 plain weave	BN/C		ORNL
CVI SiC	Nicalon satin weave	SiC/C Multilayers		Hyper-Therm
CVI SiC, Enhanced	CG- & Hi-Nicalon plain weave	C	With and w/o CVD SiC	DuPont Lanxide
CVI SiC, Enhanced	CG- & Hi-Nicalon plain weave	BN		DuPont Lanxide
Alumina	Nicalon satin weave	SiC over BN		DuPont Lanxide
Si-N-O-C	Nicalon satin weave	proprietary		Dow Corning
CVI SiC	Nicalon plain weave	nonoxidizable		Amercom
CVI SiC	Nicalon satin weave	pseudo-porous SiC		Hyper-Therm
FCVI SiC	Nicalon plain weave	C/Mullite/C		ORNL
FCVI SiC	Nicalon plain weave	C/TiO_2-$A1_2O_3$/C		ORNL
FCVI SiC	Nicalon plain weave	Porous SiC/C		ORNL
FCVI SiC	Nicalon plain weave	Porous SiC/BN		ORNL
FCVI SiC	Nicalon plain weave	Si-doped BNC-doped BN		ORNL
Alumina	Nextel 610 satin weave	BN/SiC		DuPont Lanxide

Table 1. Incomplete listing of composites evaluated in Task 2

CFCC NEWS

This newsletter is intended to facilitate communication among industrial, government, and university researchers working on the CFCC Program.

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http://www.ms.ornl.gov/cfcc/

Calendar

CFCC Working Group Meeting.
October 6–8, 1997, Harrah's Lake Tahoe, Lake Tahoe, Nev.
The focus will be on use of CFCCs in industrial applications. Industrial presentations on advances in the fabrication of CFCC materials will be held on the first day. It will be followed by workshops on key enabling CFCC technologies and materials joining, which will be conducted in collaboration with the DOE Office of Basic Energy Sciences. For registration information, contact: Shirley North. Telephone (423) 574-8860.

Materials Research Society 1997 Fall Meeting.
December 1–5, 1997, Boston Marriott/Westin Copley Place and Sheraton Boston Hotel, Boston, Mass.
For registration information, contact: Materials Research Society, 9800 McKnight Rd., Pittsburgh, PA 15237-6006.
Telepone (412) 367-3003. Fax (412) 367-4373.
E-mail: info@mrs.org

Composites, Advanced Ceramics, Materials & Structures
22nd Cocoa Beach Conference and Exposition
January 20–24, 1998, Howard Johnson/Hilton Hotels, Cocoa Beach Fla.
The conference provides a forum for non-restricted papers in broader topical areas. It will highlight an International Symposium on Advanced Synthesis and Processing. Contact: http://www.acers.org/

22nd Conference on Composites, Materials and Structures
January 26–30, 1998, Cocoa Beach Hilton, Cocoa Beach Fla.
Note: This session is ITAR-restricted to U.S. only.
For registration, contact: Management Resources, Inc., (703) 352-2211.