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A COMMUNICATION OF THE CONTINUOUS FIBER CERAMIC COMPOSITE PROGRAM

Combines Two Processing Methods

Textron's Winder

By Steve DiPietro, Textron

By combining the most attractive and cost-effective aspects of two somewhat distinct processing methodologies—slip casting of ceramics and filament winding of composite preforms—Textron has developed a novel CFCC fabrication technique.

Textron's specially modified four-axis winder can wind CFCC components up to 12 feet long, 5 feet in diameter

The technique utilizes a four-axis filament winder originally purchased for the winding of very large carbon composite components, such as rocket motor billets and thrusters.

It is a specially configured unit with an overhead creel delivery system and is one of the larger filament winding units in the United States.

The winder was modified for the CFCC Program to wind CFCC preforms using either ceramic monofilaments or tows. This modification necessitated fabrication of specialized mandrel assemblies and slurry delivery hardware that allow simultaneous impregnation of ceramic slurries onto fiber tows or filaments as a preform is being constructed.

The machine is computer controlled and may be programmed off-line or at the winder itself. All four axes of motion are independently programmable, thus providing essentially infinite capability to wind complex geometries and fiber architectures.

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Inside

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Textron Winder

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"Combining the best elements of filament winding and traditional slip casting enabled development of a cost-effective and very scalable CFCC fabrication process in much more rapid fashion.

The four-axis winder can wind CFCC components up to 12 feet long and 5 feet in diameter. Winding angle possibilities are unlimited, and components up to 3000 pounds can be accommodated on the headstock/ tailstock mandrel mounting hardware.

Combining the best elements of filament winding and traditional slip casting enabled development of a cost-effective and very scalable CFCC fabrication process in much more rapid fashion than if we attempted to develop a new processing approach from the "ground up."

Textron has been using the filament winder principally to fabricate CFCC immersion burner tubes and combustor cans. The company now is beginning to get involved in other commercial activities to fabricate CFCCs that will be evaluated in various high-temperature processing applications.

Net-Shape Fashion

Slip casting of the CFCC green body as it is being filament wound maximizes matrix density and permits formation of the preform geometry in totally net-shape fashion. No pressure is required to effect matrix densification using a slip casting–based process.

Computer-controlled filament winding of nitride-bonded silicon carbide CFCC parts using ceramic monofilaments or tows confers essentially infinite capability to wind different reinforcement architectures and allows Textron to tap into a huge technology base to fabricate CFCC preforms.

Four Independent Axes

The significance of TSM's filament winder itself principally relates to its size and flexibility. The McClean-Anderson winder is capable of winding CFCC parts up to twelve feet long and six feet in diameter with four computer-driven independent axes of freedom.

The importance of the filament winding process for CFCC preform fabrication is in process speed, simplicity, and efficiency.

Because Textron uses a modified version of ceramic slip casting to predensify the ceramic matrix (without pressure) and form the preform shape, no expensive restraint tooling or fixturing is needed.

Tooling is fabricated from low-cost, yet reuseable, materials (such as plaster and aluminum) that can be quickly and easily procured and assembled. Since the fabrication process is totally net-shape, no machining of the preform is required at any step in the processing cycle. This also significantly lowers manufacturing costs.▲

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O'Leary & CFCC

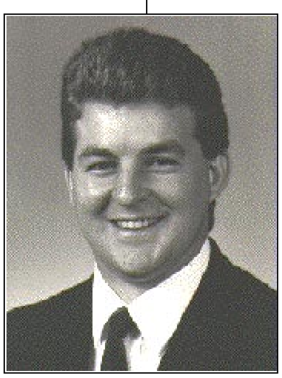
Hazel O'Leary, Secretary of Energy in the U.S. Department of Energy, at the 1st Industrial Energy Efficiency & Exposition in Washington, D.C., on May 1, 1995. She is holding a CFCC combustor plate and talking to Leon Ostrowski, Composites Business Development at Dow Corning about the benefits of using CFCCs in U.S. Industry. Andy Szweda, CFCC Program Manager, is in background.

Integrated Effort

Industry and Government are Working Together

By Rick Lowden, Oak Ridge National Laboratory

Technical and economic advances in the development and fabrication of materials for industrial applications must be supported by an integrated effort that combines the experience and facilities of industry with the expertise and specialized talents available at universities and national laboratories.



Rick Lowden

The CFCC Program was structured to take advantage of the valuable resources available in all segments of the community to more rapidly overcome the challenges involved in the development of these advanced materials for industrial and power- generation applications.

Industry has the lead role in the CFCC Program with teams of fabricators and users working together to determine the appropriate materials and processing routes for selected applications.

Universities and government laboratories have assumed a supporting role, conducting the more basic or generic studies such as composite design, materials characterization, test method development, and the investigation of performance-related phenomena.

A Challenge

Integrating the activities of the industrial teams and those in academia and national laboratories has been a challenge. Recent decisions have resulted in a streamlining of the process to establish small research projects to better serve the needs of the industrial teams.

From the beginning of the CFCC Program, the nature of the interaction between the industrial partici-

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" From the beginning of the CFCC Program, the nature of the interaction between the industrial participants and the supporting technologies efforts has been a concern.

dently of the other tasks. To accomplish the goals of the task, model or representative composite systems have been employed to examine effects and phenomena applicable to a broad range of materials, processing techniques, and service environments.

Developing Materials

Fundamental issues are to be addressed, models developed, and mechanisms and trends examined. The products of these endeavors will be used in performance simulations and life prediction and will act as guides for the development of reliable composite materials and components.

The projects are not to be "service work" but are to be independent efforts in themselves, developing techniques and methodologies, computer codes, and facilities and equipment in support of the processing and fabrication tasks.

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Working group meetings, conferences, and publications have been the chosen media by which interaction between the tasks has occurred. Surveys and personal discussions have also been used to disseminate information, to prioritize efforts, and to gain input into the needs of the industrial teams.

There are many alternatives to meetings, presentations, and publications for interaction between participants. The options include, but are not limited to, user agreements, cooperative research and development agreements, and work for others. User agreements encompass working with the High Temperature Materials Laboratory (HTML) centers at ORNL. The process to become a user is straightforward and has been refined to make access to the HTML relatively easy. Work proposals between the potential user and the appropriate center are developed.

The proposals are reviewed by a panel for acceptance. If the results of the interaction are to be published openly, then there is no cost (assuming all other requirements are met). If the work is to be proprietary, there are modest fees and a proprietary information agreement (PIA) must be negotiated. Note that PIAs involve lawyers, adding considerable time and mental anguish to the process.

Another Avenue

Another avenue for cooperation is the cooperative research and development agreement (CRADA). Typically, CRADAs involve proprietary information; thus, again, a PIA is necessary. In most cases costs are shared, meaning either funds in from the company or in-kind effort, and support from a sponsor for the supporting technologies participant(s).

Work for others (WFO) is another method by which an organization can gain access to the personnel and facilities at the national laboratories. The services of the labs are purchased, and as with CRADAs, this requires a lengthy paper trail.

Progressing

The processing and applications tasks of the CFCC Program have progressed well with many successes and advancements, and thus the Program continues into a second phase. The initial portion of the industrial efforts focused on applications, material selection, and process development.

As projects are now transitioning into process engineering and component development, the needs of the teams are changing. Supporting Technologies has been structured to follow the progress and evolving requirements of the processing tasks.

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Efforts in Task 2 originally emphasized the fundamental aspects of the design of composite materials (e.g., mechanical modeling and interface studies). As the program matures, efforts are shifting toward the design with composites, with projects in the areas of test methods and performance-related phenomena gaining in importance.

Transition Period

Task 2 is in a transition period. Mechanical models have been incorporated into computer codes. Techniques for the characterization and testing of composites have been developed or refined. Standardized test methods have been submitted to ASTM for acceptance.

Instruments and systems such as the Interfacial Test Systems and the Thermal Conductivity Microprobe have been designed, built, and tested. These are the products of Supporting Technologies, and they are ready to be utilized. The time for interaction between the tasks is at hand.

The importance of interaction and support within the CFCC Program was emphasized by the many discussions at the working group meeting in Bozeman. Dave Richerson recognized the need for better cooperation between tasks and took it upon himself, with approval from the Program office, to suggest methods to foster interaction.

A meeting was held at Oak Ridge National Laboratory in December 1994 to review Dr. Richerson's recommendations. Representatives from the Department of Energy Chicago and Oak Ridge Operations offices and the Supporting Technologies task were in attendance. It was determined that the industrial teams had considerable interest in interaction with Task 2, especially in the areas of testing and characterization. However, the paperwork and effort required to initiate the interaction was burdensome. Too much was involved in establishing property rights, funding, etc., and time is a critical issue.

Simplifying

An on-the-spot decision was made to simplify the process by which small short-term projects could be initiated, therefore permitting rapid access to Task 2 facilities and personnel by the industrial teams.

Once a proprietary information agreement (PIA) between the appropriate organizations is in place, all that is necessary is a short proposal from the participants describing the tasks and schedule. The proposals are quickly reviewed, and once the proposal is accepted, funding is transferred to the Task 2 participant to conduct the work. All information is kept as proprietary unless other arrangements are agreed upon by the participants. The projects are to be small and short, typically < $30K and two months in length.

The purpose is to enhance the interaction between the industrial programs and the supporting technologies tasks. Many of the industrial teams require limited but specialized testing or characterization of their materials to guide changes in processing or composition and to accelerate their progress.

New Initiatives

Jill Jonkouski, of the Chicago Operations Office, pursued the opportunity with fervor. Her efforts have resulted in the establishment of three ongoing projects, with others being negotiated. For example, Alzeta is utilizing the Materials Characterization User Center of the HTML for postservice characterization of burner component candidate materials.

Textron Specialty Materials is working with the Physical Properties User Center of the HTML to study the thermal diffusivity and conductivity of their composite materials.

The Materials Characterization User Center in the HTML is assisting in the characterization and testing of Amercom's advanced interface concepts. A proposal between Dupont Lanxide Composites and the HTML Mechanical Properties User Center for composite testing and characterization is being negotiated.

In addition, arrangements are being made for Argonne National Laboratory to conduct the nondestructive characterization of CFCC combuster

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A Message from the Program Manager

I would like to take this opportunity to introduce myself to those of you who I have not had the chance to meet. I have officially been the program manager since April, and I look forward to meeting many of you at the CFCC fall meeting September 18 and 19 in Chicago.

I want to thank everyone who participated in the first Office of Industrial Technologies Symposium and Exposition. We had an outstanding turnout for this event and most would probably agree that it was a great success.

The CFCC team had a table to attract the attention of Secretary O'Leary in the exhibit hall and give her a brief explanation of what the program was all about.

As you all know, the FY 1996 budget battle has been fierce (and at this writing, is not over yet). Everyone has done a terrific job of getting the word out on the many benefits of the program. Your efforts have been greatly appreciated. Even though things are looking good for FY 1996, the FY 1997 budget campaign will likely be an even greater struggle.

Now that we are into Phase II, I am looking forward to seeing many of you demonstrate components in the field and am anxious for our first commercial success.

I am sure many of you are aware of OIT's reorganization that took place at the beginning of the year. I think the CFCC Program fits well in this new organization as a crosscutting technology.

Technologies and components of the CFCC program support many of the seven industries (aluminum, chemicals, glass, forest products, metal casting, steel, and petroleum refining) on which OIT is focusing.

In closing, I would like to say that I look forward to working with all of you and am eager to do anything I can to ensure the success of the program.

Please feel free to contact me anytime. My phone number is (202) 586-3646, and I can be reached through the Internet at merrill.smith@hq.doe.gov.

Merrill Smith

Merrill Smith, right, the new DOE CFCC program manager with Jim Wessel. We want to congratulate Jim Wessel on his retirement from Dow Corning Corp. Jim has worked on the CFCC program since its existance, and we appreciate his hard work and the guidance that he has provided.

Dow Corning's New Application

Tube Hangers

New CFCC Components for Crude Oil Furnaces

By Andy Szweda,
Dow Corning

Dow Corning is working with Chevron Research and Technology Company on a new application in its CFCC Program—a tube hanger for use in a crude furnace at Chevron's Richmond, California, refinery.

The first step in refining crude oil at any refinery is to heat the oil in a crude furnace to break it into its various distillates, which are then further refined to provide products such as gasoline, jet fuel, fuel oil, and kerosene.

The crude furnace consists of a fire-box, which is refractory lined to insulate the steel firebox skin, coils of steel or alloy tubing through which the crude oil is transmitted and heated, and the metal tube hangers that support the furnace tubes in the firebox (Fig. 1).

Most crude furnaces are heated by natural gas and operate in the 1500–2000°F range. A typical firebox environment contains mostly nitrogen, carbon dioxide, and carbon monoxide along with smaller amounts of oxygen, water vapor, and sulfur dioxide.

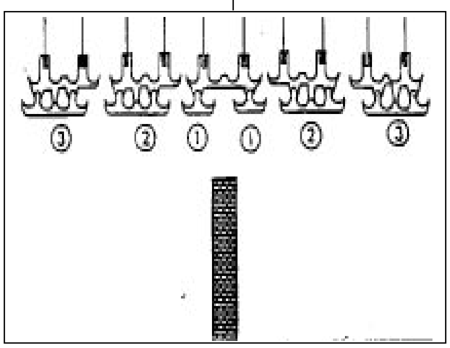

Fig. 1. Pipe hanger configuration in crude unit furnace.

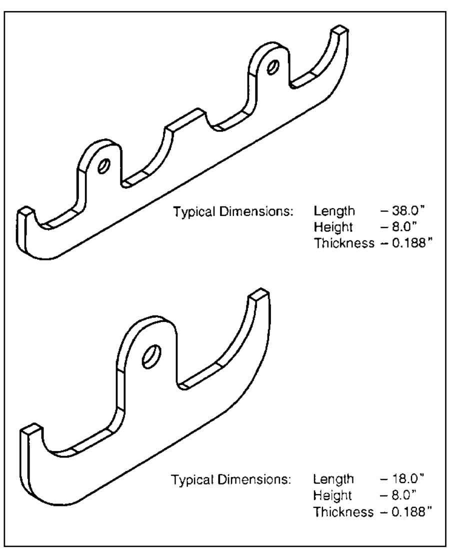

Fig. 2 (bottom). Design of CFCC pipe hangers.

Operating these furnaces at higher firebox temperatures (2000°F rather than 1500–1600°F, for example) allows increased crude throughput, which results in higher production for the refinery.

However, operating at these higher temperatures causes premature creep failures of the metal tube hangers now being used. Thus, these higher temperatures cannot be maintained for long durations.

By using CFCCs capable of withstanding high temperatures, it is expected that tube hangers can be made to last longer, resulting in increased production and profit along with reduced NOx emissions and downtime.

Dow Corning will fabricate two types of CFCC tube hangers (Fig. 2) for installation in a crude furnace at Chevron's Richmond refinery.▲

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Solar Turbines

CFCC Design Target: Lean Premix Combustors

By Jane Simpson,
Solar Turbines

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Successful retrofitting of hot section components with ceramics will result in gas turbines with improved performance, lower emissions, and greater temperature durability.

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Since they are relatively flaw insensitive, even the thirty inch diameter outer combustor liner can be fabricated in one piece.

Solar Turbines participates in several ceramics programs that support the DOE’s mission of national energy conservation and environmental preservation.

Successful retrofitting of hot section components with ceramics will result in gas turbines with improved performance, lower emissions, and greater temperature durability. Because hot wall combustor liners are generally exposed to only minimal mechanical stresses, CFCCs are emerging as strong contenders for the combustor application.

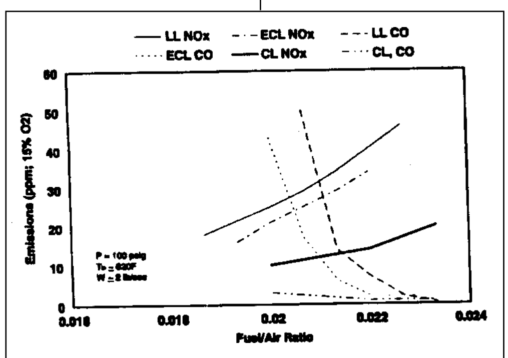

Fig. 1. Comparison of CO levels in metal and ceramic liners.

Solar’s primary design targeted for CFCCs is an annular, lean premix combustor. CFCC liners for this application are being analyzed, designed, and fabricated under the DOE–sponsored Ceramic Stationary Gas Turbine (CSGT) program. In the current configuration, CFCCs will replace an 8-inch-long cylindrical portion of the 13-inch-diameter inner wall and 30-inch-diameter outer wall of the combustor.

Advantages

CFCCs potentially offer many advantages in the combustor application. Because they are relatively flaw insensitive, even the 30-inch-diameter outer combustor liner can be fabricated in one piece. Also, their toughness gives them the ability to withstand impacts during handling, assembly, and operation. Another advantage of CFCCs is that failures tend to be noncatastrophic, which minimizes spalling or separation of large pieces and the potential for damaging monolithic ceramic and/or metallic parts downstream. In addition, the fiber architecture of CFCCs can be tailored to give strength and stiffness in the directions needed. Finally, CFCCs have the high temperature capability needed to endure the CSGT target turbine rotor inlet temperature of 1121°C (2050°F).

Solar is considering several CFCCs for the combustor liner application. Subscale liners approximately 8 inches long and 8 inches in diameter will be tested in a rig that simulates the engine combustion environment.

The test is controlled so that the maximum wall temperature of the CFCC is 1177°C (2150°F). Under the CSGT program, the subscale test plan includes 1-, 10-, and 100-hour tests on DuPont Lanxide’s (DLC) Enhanced SiC/SiC and Babcock & Wilcox’s (B&W) Alumina/Alumina. (DLC and B&W are team members and subcontractors on the CSGT program).

Nondestructive evaluation is performed at Argonne National Laboratory (ANL) on each liner before and after every test. ANL, another subcontractor on the CSGT program, has developed several methods for nondestructive evaluation of CFCCs. The methods being studied at ANL include thermal diffusivity mapping, multiscan X-ray–computed tomography, and single wall film X-ray.

Ultrasound

Air-coupled ultrasound at QMI in Costa Mesa, California, is also being evaluated for CFCCs. Pretest results on one of the DLC liners shows excellent correlation between the results of thermal diffusivity mapping, X-ray–computed tomography, and water-coupled ultrasound (performed by DLC).

Mechanical properties tests and metallography will be performed on the liners after the rig tests are complete. These data will be correlated to ANL's results.

One-hour and 10-hour steady state tests were successfully completed for the DLC SiC/SiC.

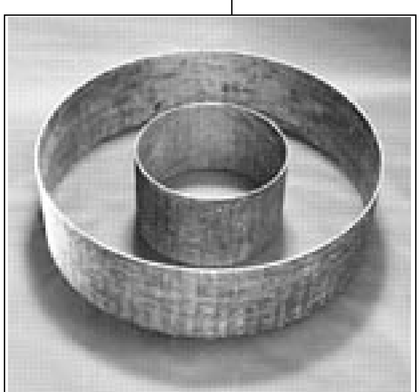

Fig. 2. Full scale 3D woven SiC/SiC liners fabricated by B.F. Goodrich Supertemp.

Nondestructive evaluation using thermal diffusivity mapping indicates growth of low-density regions after the 10-hour test. A 1-hour test of the B&W alumina/alumina was also completed successfully. Nondestructive evaluation data for this liner are not available at this time.

As part of a study to document the role ceramics can play in lowering NO_x emissions, Solar conducted a series of combustor rig tests comparing metallic and ceramic combustors. Two cylindrical metallic combustors (8 inch diameter) and a cylindrical CFCC combustor were all tested at typical gas turbine operating conditions in a subscale test rig. The same natural gas, premixing fuel injector was used for all of the testing.

The metallic combustors differed in the amount of cooling air used to maintain acceptable liner temperatures. A conventional louvre-cooled liner used the most liner cooling air. An effusion-cooled liner used less cooling air. Only backside cooling was used with the ceramic liner.

As shown in Fig. 1, at a given NO_x level the metal liners showed higher CO levels than the ceramic liner. This is attributable to the quenching effect of the liner cooling air. With no cooling air injection, the ceramic combustor produced NO_x levels below 10 ppm (15% O_2) with low CO emissions. The two metallic liners were limited to NO_x emissions near 20 ppm without producing excessive CO.

Currently these preliminary data are being verified. A significant technical challenge remains in transferring these rig test results to a full-scale gas turbine combustion system.

Full-scale testing of CFCCs will begin in late spring/early summer. Solar received two sets of full-scale 3D woven SiC/SiC liners fabricated by B.F. Goodrich Supertemp in March 1995 (Fig. 2). After proof testing in atmospheric and high-pressure test rigs, one of these sets will be tested in the CSGT engine rig. Upon successful completion of these tests, a ceramic combustor liner will be installed at a customer site for a 4000-hour field engine test.▲

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Pulp & Paper

By James Weddell,
Du Pont Lanxide Composites Inc.

> “Representatives of the two companies said that if CFCCs could significantly increase component longevity, they are interested in pursuing the discussions and testing CFCC products.

Replacing conventional materials with new, light-weight, high-temperature-resistant CFCCs may solve a variety of equipment problems for the pulp and paper industry.

> CFCCs may solve a wide variety of equipment problems for the pulp and paper industry.

That is the message that representatives of DOE’s CFCC Program passed on during recent visits to two of the industry’s largest corporations.

Discussions with the James River Corporation and the Chesapeake Forest Products Corporation, both headquartered in Richmond, Virginia, centered on using CFCCs for components such as water-cooled tubes and spouts in smelting operations, fire tubes, paper hangers, burner boxes, and spray nozzles.

These and other devices used to manufacture pulp and paper are particularly prone to wear and to heat-caused deterioration. Representatives of the two companies said that if CFCCs could significantly increase component longevity, they are interested in pursuing the discussions and testing CFCC products.

Representing the CFCC Program for the visits were Scott Richlen and Merrill Smith, DOE; Bill Long and Rich Goettler, B&W; Jim Bird, Amercom; Jim Wessel, Dow Corning; Dave Magley, Dow Chemical; Bruce Thomson and Dick Krutenat, Textron; and Jim Weddell, DLC.

Following is a report of equipment problems and possible CFCC applications discussed with the two companies.

JAMES RIVER

Smelt Floor

The smelt floor is made of 2.5- to 3-inch-diameter water-cooled tubes that conduct 1600°F molten smelt, sometimes reaching 2000°F. The carbon-steel-studded tubes are coated with a high-alumina base refractory coating that chemically corrodes in one to six months. If the molten smelt contacts water inside the tubes, a steam explosion results, which occurs a few times each year.

Smelt Spout

Molten smelt from the smelt floor flows out of the furnace and down the 3-foot-long, 5-inch-wide, carbon steel, water-cooled spouts. The spouts are subject to both erosion and corrosion problems that create conditions under which steam explosions can result. It can take two days to replace a spout, which costs a large mill $500,000 in lost production. If the spouts could be made to last longer, the mills would save money.

Paper Driers

James River dries its tissue on 15-foot-long, 18-foot-diameter drums using steam heated by natural gas. The drums are made of Corten metal. The company would like to find a way to use higher temperatures in the process. Their representatives suggested that CFCC Program officials discuss the possibilities with ABB Montreal Technical Center, which supplies the equipment.

Evaporators
James River uses stainless steel equipment reaching a maximum of 270°F in the black liquor area. The company has a metal wastage problem resulting from erosion and/or corrosion, both of which might be allayed by utilizing CFCCs.

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Liquor Heaters
These heaters have 1-inch-diameter, 30-foot-long tubes containing a high-velocity NaOH/Na_2S solution at 300-320°F. The tubes must be resistant to thermal expansion and warpage so that they can easily be extracted from the shell. Exotic steel alloys such as Inconel are now being used.

Blow Tank Targets
After wood chips are partially digested, a slurry is shot at the targets to break them up. The equipment is subject to erosion problems.

CHESAPEAKE

The CFCC group toured the area containing recovery boilers where black liquor is injected into a furnace to burn carbon and recover sodium salts.

A corrosive solution is sprayed out of multiple pipes into the yellow-hot furnace. Solids drop to the furnace floor, burn, and flow out through smelt spouts. Shielded workers continually ram metal rods at the smelt spouts to keep the smelt flowing.

Burner Components
The ends of these components in the recovery boilers burn off regularly. They currently use carbon or stainless steel.

Black Liquor Spray Nozzle
These cost about $800 each and are used to spread the black, 700°F liquor spray into the furnace duct areas. The carbon steel component is subject to corrosion.

Dust Collector Cones
These are 8-inch-diameter cylinders inside 10-inch-diameter cylinders. A combination of air and dust swirls in the annulus. Erosion problems occurring at relatively low temperatures result in the company replacing about 25 of them each year.

Pulverizer Fans.
Made of carbon steel, these are replaced about once a year. The company tried ceramic block liners, which lasted as long as 8 years.

Sawdust Cyclone
Sand in the sawdust erodes the cyclone, which might last longer if it were made of CFCC materials. The results of erosion requires that the company replace sections every few years.▲

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Supporting Technologies

Damage is Major Concern

Thermal Shock

By Raj N. Singh, James E. Webb, Hongyu Wang, and Umashankar Anadakumar, University of Cincinnati

Thermal shock damage is a major concern for ceramic materials for use in high-temperature applications. The development of CFCCs for such severe service conditions requires an understanding of the nature of thermal shock damage because of the increased susceptibility of damaged components to environmental attack.

Also, thermal cycling, and therefore multiple thermal shocks, in these materials leads to 'thermal fatigue' damage. Nondestructive evaluation (NDE) techniques to detect thermal fatigue damage is essential to monitor the structural integrity of real components.

Thermal shock studies to characterize damage and evaluate the applicability of NDE techniques to detect damage are being conducted at the University of Cincinnati in collaboration with William Ellingson at Argonne National Laboratory, Rick Lowden and Peter Tortorelli at Oak Ridge National Laboratory, and Andy Szweda of Dow Corning.

Approach

In this study, thermal shock damage in CFCCs was induced by a simple water quench technique followed by both destructive and nondestructive techniques to characterize the damage. The quench technique involved heating test bars to various temperatures, in a tube furnace, followed by a free fall into a water quench bath as shown in Fig. 1. Four-point bend tests on these bars after quenching were used as a destructive method to determine critical properties such as matrix cracking strength, ultimate strength, and work of fracture. NDE techniques were used to measure Young's modulus and thermal diffusivity.

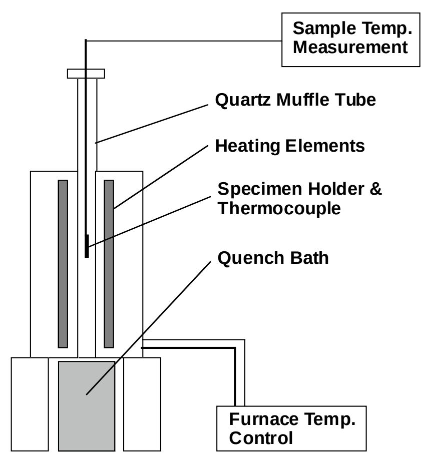


Fig. 1. Apparatus for the thermal shock study by the water quench technique.

Three composites were evaluated in this study. A Nicalon™ fiber with a chemical vapor infiltration (CVI) SiC matrix and a Nextel™ fiber with a CVI SiC matrix were supplied by Oak Ridge National Laboratory. A Nicalon™ fiber with a polymer impregnation and pyrolysis (PIP) SiC matrix was supplied by Dow Corning Corporation. Three different fiber architectures, uniaxial alignment, alternating 0/90° layers, and 2-D woven fibers were also studied.

Destructive Evaluation

The composite and fiber geometry that demonstrated the best thermal shock resistance was the 2-D Nicalon™ CVI SiC. Figure 2 shows the stress displacement behavior for this composite before and after two thermal shock tests. The strength of this composite is shown in Fig. 3 as a function of the quench temperature difference, ΔT (difference in temperature between the furnace and water bath). No significant degradation of the ultimate strength was present below ΔT = 700°C. This ΔT, below which no significant damage occurs, is called the critical temperature difference ΔT_C. Increasing ΔT to 1000°C results in only a 10% decrease of the ultimate strength. Retaining 90% of the ultimate strength in this CFCC is an impressive result when compared to most monolithic ceramics that exhibit a catastrophic drop in strength once ΔT_C is exceeded, as shown by data for a hot pressed SiC in Fig. 3. These results demonstrate that the water quench technique in combination with strength tests can be used to induce and monitor thermal shock damage in CFCCs.

Nondestructive Evaluation

Obviously, determination of the ultimate strength is an essential but also a destructive characterization technique that cannot be used in service. Young's modulus, determined from the resonant frequency of a composite bar after being struck by a small hammer (Grindosonic method), can be obtained multiple times with no resultant damage.

Figure 4 shows these modulus measurements as a function of ΔT for the 2-D Nicalon™ CVI SiC composite. Young's modulus shows a steady decrease with increasing ΔT. Above ΔT = 800°C there is also a sharper decrease in modulus that is in a similar temperature range to the small drop observed in the ultimate strength in Fig. 3.

More appropriate use of this NDE technique was made by measuring Young's modulus as a function of the number of quench cycle for a single specimen. This was done for two composite bars, one cycled at ΔT = 600°C (below the ΔT_C), the second at ΔT = 800°C (above the ΔT_C), with the results shown in Fig. 5.

The modulus for both specimens is essentially identical before quenching. After a single quench cycle, the decrease in modulus is similar for the two temperature differences. However, with increasing numbers of quench cycles, the specimen shocked at ΔT = 800°C shows a significantly larger decrease in the modulus, retaining less than 65% of the original value after 14 cycles. After 14 cycles at ΔT = 600°C, more than 90% of the original modulus is retained. Therefore, as ΔT is increased, the degrada-

Fig. 2. Stress-displacement curves of the 2-D Nicalon™ CVI SiC composite before and after quenching.

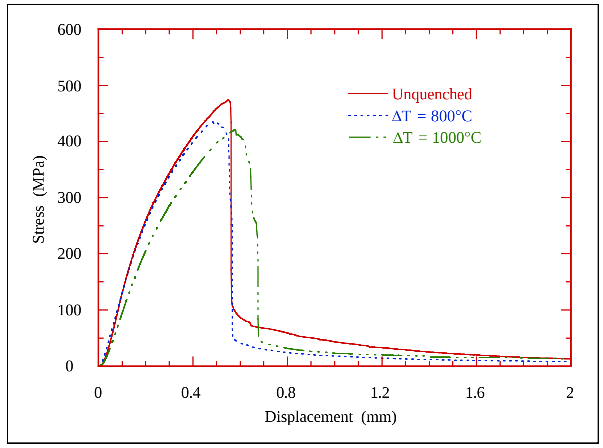


Fig. 3. Influence of quenching temperature (ΔT) on the ultimate strength of a 2-D Nicalon™ CVI SiC composite and a monolithic hot-pressed SiC.

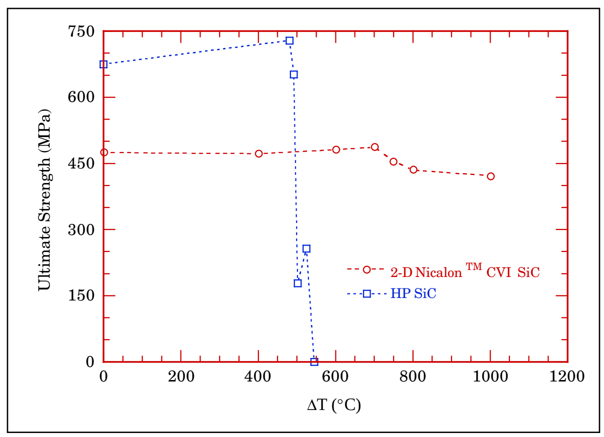
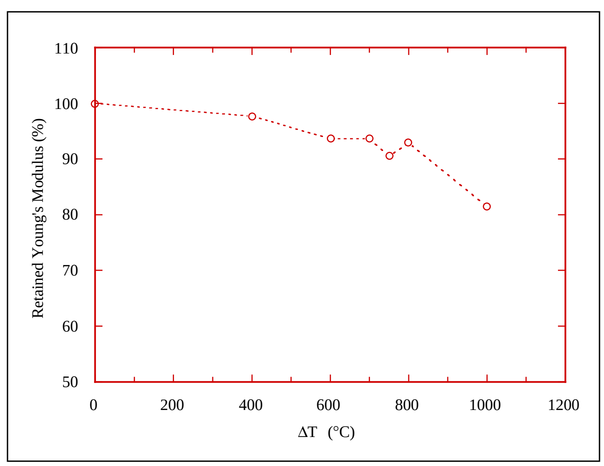


Fig. 4. Effect of quench temperature difference on Young's modulus of the 2-D Nicalon™ CVI SiC composite.



tion due to thermal fatigue becomes more severe.

Another NDE technique of measuring thermal diffusivity was used by William Ellingson at Argonne National Laboratory to characterize thermal shock damage in another composite (2-D Nextel™ CVI SiC). Figure 6 displays this information as a function of ΔT. A rapid decrease in the thermal diffusivity is seen at ΔT = 800 and 1000°C. Thermal diffusivity measurements in the same composite material after 0, 1, and 4 quench cycles at ΔT = 800°C are shown in Fig. 7.

The thermal fatigue effect shows a decrease in thermal diffusivity with increase in the number of quench cycles. It is apparent from these results that both NDE techniques can be used to monitor damage in CFCCs due to thermal shock.

Damage Mechanisms

The three characterization techniques discussed, four-point bend, Young's modulus, and thermal diffusivity, essentially measure the effect of thermal shock damage on various material properties. Optical microscopy was employed to search out the nature of this damage.

Figure 8 is a representative micrograph after 10 quench cycles at ΔT = 600°C. In this micrograph, the light regions are the CVI SiC matrix, the darker oval shapes are the Nicalon™ fibers, and the large black region in the center is a typical void between four fiber bundles.

Thermal shock damage in the form of matrix cracks emanating from three corners of the large central void was produced after one thermal cycle. After 10 quench cycles no significant lengthening of the initial cracks was observed; however, these cracks did appear to widen, and some fibers were removed indicating damage to the fiber-matrix interface. At ΔT = 800°C optical microscopy showed more extensive matrix cracking and fiber-matrix

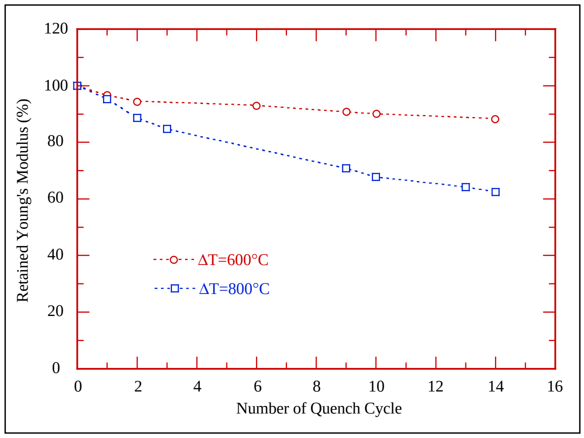


Fig. 5. Effect of cyclic quenching on Young's modulus of the 2-D Nicalon™ CVI SiC composite.

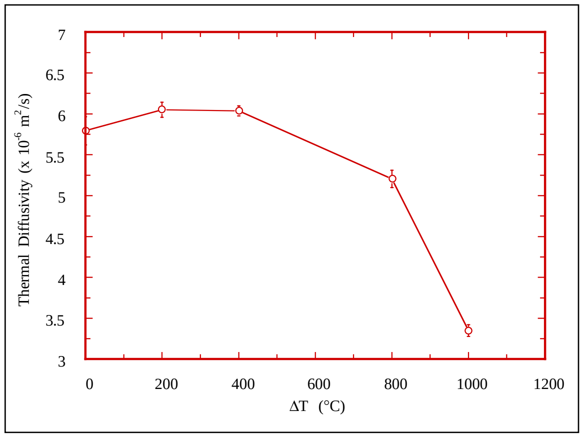


Fig. 6. Effect of quench temperature difference (ΔT) on thermal diffusivity of the 2-D Nextel™ CVI SiC composite.

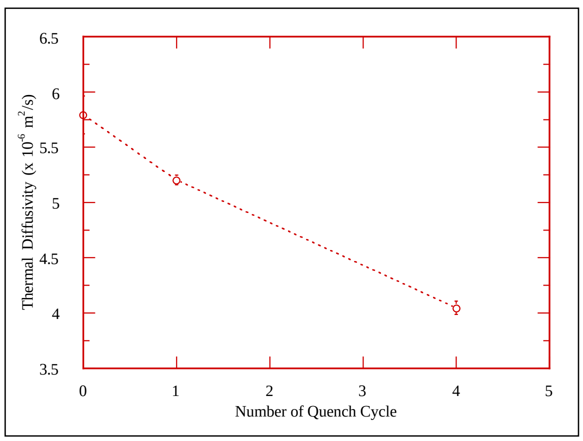


Fig. 7. Effect of cyclic quenching on thermal diffusivity of the 2-D Nextel™ CVI SiC composite at ΔT = 600°C.

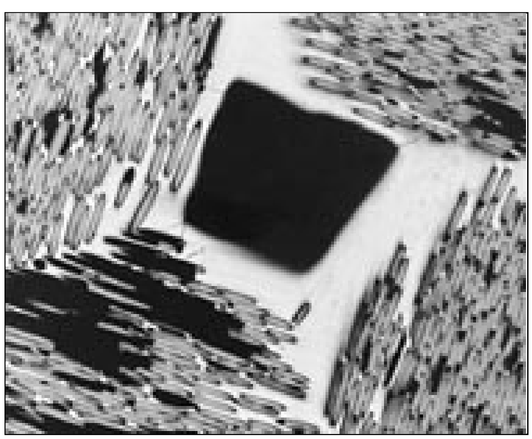


Fig. 8. Optical micrograph after 10 quench cycles at ΔT = 600°C.

interface damage as well as extension and formation of new cracks with multiple quenches. These microscopic observations support the more severe effect of cyclic thermal shock at higher ΔTs shown by Young's modulus measurements.

It is clear that thermal shock is responsible for the formation of cracks in the matrix. However, the susceptibility of the fiber-matrix interface to oxidation suggests that oxidation and thermal shock could both be responsible for damage to the fiber-matrix interface. Studies by Peter Tortorelli, at Oak Ridge National Laboratory, of oxidation effects in combination with thermal shock damage will help distinguish between these two damage mechanisms.

Summary

A methodology is developed to induce and characterize thermal shock damage in CFCCs. This method employed the water quench technique to successfully generate thermal shock damage which was characterized by destructive and NDE techniques on a variety of materials. The superior thermal shock resistance of CFCCs was demonstrated by a minor drop in the ultimate strength for quenches above ΔT_C. The clear variance of Young's modulus and thermal diffusivity with both ΔT and number of shock cycles supports the feasibility that these NDE techniques can monitor thermal shock damage in real components.

Future Directions

Further work will be conducted to determine the effect of thermal shock damage on oxidation resistance. Continuing optical microscopy studies will be aimed at understanding the mechanisms of mechanical damage of samples before and after thermal shock.

Additional tests will be done to study the mechanical fatigue properties of these composites after subjecting them to thermal shock damage. Influence of the thermal shock damage on the high temperature mechanical properties will also be investigated. The use of NDE techniques for characterizing damage due to the mechanical stress and thermal shock will continue to be evaluated.▲

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Integrated Effort with Industry

Continued from page 3

liners prior to and after exposure to simulated service environments.

As previously stated, the purpose of these small projects is to enhance the interaction between the industrial programs and the supporting technologies tasks and to better assist the industrial participants in the testing and evaluation of individual and specific materials systems.

The services provided may be material, process, and application specific and thus necessitate close interaction between the industrial teams and support personnel. The task will focus on providing direct technical support and services to the manufacturers and users. Interaction may continue to be accomplished in the form of direct services, standardized testing, or CRADAs.

The statement "We don't do service work!" may have become passe. Let us not think of these efforts as service work but as relationships that are the key to the success of the CFCC program.▲

Help! Lost Box

At the recent First OIT Industrial Energy Efficiency Symposium & Expo at the Washington DC Renaissance Hotel, Dow Corning lost a box containing CFCC demonstration samples and literature.

If anybody has mistakenly received this box or has any information on it's whereabouts please notify Andy Szweda, Dow Corning Corp., Tel: (517) 839-0817, E-mail: usdccp63@ibmmail.com

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The Supporting Technologies Task of the CFCC Program will have initial responsibility for conducting the survey and compiling the results.

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Anyone with questions and comments about this effort, or with suggestions of facilities to be included, should contact Peter Tortorelli, ORNL, by phone (615-574-5119), FAX (615-574-5118) or E-mail (pft@ornl.gov).

Environmental Test Facilities

Survey to Begin

By Pete Tortorelli, Oak Ridge National Laboratory

One of the important aspects related to the use of ceramic composites in industrial applications is the aggressiveness of many of the environments to which these materials are subjected during service.

Because of this, the corrosion and erosion performance of CFCCs is a key issue facing materials producers developing ceramic composites for targeted applications.

Often corrosion behavior is assessed using built-for-purpose rigs at various research facilities or by arrangements between material producers/fabricators and specific users, but a technical community–wide awareness of the existence and availability of environmental test facilities appears to be lacking.

A general survey of existing test facilities, availability, and protocols will be conducted for use by those involved with ceramic composites.

To address this deficiency and to encourage testing to (1) examine the suitability of various CFCCs for industrial service and (2) produce the information needed for further development, a general survey of existing test facilities, availability, and protocols will be conducted for use by those involved with ceramic composites.

It is expected that the survey will not only include relevant facilities of organizations involved in the various tasks of the CFCC Program, but also those associated with NASA, DoD, and universities and industrial concerns.

The Supporting Technologies Task of the CFCC Program will have initial responsibility for conducting the survey and compiling the results. The process is viewed as ongoing, and this Environmental Test Facilities (ETF) document will require regular updating.

To accommodate such revisions efficiently, it is anticipated that the ETF document will eventually be available on-line through the World-Wide Web.

It is planned that, for each test unit, the ETF document will contain
(1) facility description,
(2) operating conditions,
(3) specimen design considerations,
(4) availability and conditions for use (including proprietary arrangements),
(5) costs and/or other considerations (for example, in-kind work) involved with use, and
(6) name of person to contact for more information.

Individual contacts for this compilation have begun, and a survey will be sent out in July. Questions and comments about this effort, or suggestions of facilities to be included, should contact Peter Tortorelli, ORNL, by telephone (615) 574-5119, FAX (615) 574-5118, or E-mail (pft@ornl.gov).▲

MRS Symposium

100 Papers Presented at Boston Gathering

By Rick Lowden, Oak Ridge National Laboratory

A Symposium on Ceramic Matrix Composites: Advanced High-Temperature Structural Materials was held at the Fall Meeting of the Materials Research Society in Boston, Massachusetts, November 28–December 2, 1994. The symposium was organized by Richard A. Lowden and Mattison K. Ferber of Oak Ridge National Laboratory, John R. Hellmann of Pennsylvania State University, Steven DiPietro of Textron Specialty Materials, and Krishan K. Chawla of New Mexico Technological University. The symposium was sponsored by the DOE's Office of Industrial Technology's Continuous Fiber Ceramic Composites Program, the Air Force Office of Scientific Research, and NASA Lewis Research Center. Among the competing materials for advanced, high-temperature applications, ceramic matrix composites are leading candidates.

Stimulating Interaction

The objective of the symposium was to bring together researchers concerned with the various aspects of ceramic composites to stimulate interaction between the multiplicity of disciplines involved in the successful utilization of these advanced structural materials.The excellent attendance and the over 100 papers presented in the symposium demonstrated the continued interest in, and importance of, ceramic matrix composites. Many of the well-established leaders in the field were present as well as some welcome new researchers. The opening presentation described the benefits of the utilization of continuous fiber–reinforced ceramic matrix composites in industrial and power generation applications. The projected cost and energy savings and pollution reductions resulting from the insertion of these materials in only a fraction of the available systems are significant, lending great credibility to the continued pursuit and investigation of this class of advanced materials.

Papers

The symposium was organized such that papers concerning constituents, fibers, and matrices were presented first, followed by composite processing, modeling of mechanical behavior, and thermomechanical testing. More stable reinforcements are necessary to enhance the performance and life of fiber-reinforced ceramic composites and to ensure final acceptance of these materials for high-temperature applications. Encouraging results in the areas of polymer-derived SiC fibers and single crystal oxide filaments were given, suggesting composites with improved thermomechanical properties and stability will be realized in the near future.

A significant number of the presentations about matrices dealt with reduction or displacement reactions, also known as “thermite” reactions. Controlled chemical interactions between two materials, which in the described systems are typically exothermic, are used to form different, more stable “composite” compositions with fascinating microstructures. Interesting combinations of materials were explored, many having great promise for use as composite matrices and/or stand

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“Corrosion, thermal shock resistance, and cyclic thermomechanical loading were primary concerns for limiting the life of ceramic composites in many of the potential applications.

alone compounds. Continued improvements in the mechanical models, combined with more extensive testing, also supported the fundamental premise for examining continuous fiber ceramic composites (i.e., low modulus ceramic fibers can be added to stiff ceramic matrices to produce materials with good strength, high-temperature performance, and execptional toughness).

Fiber-Matrix Interfaces

Two days of the symposium were devoted to fiber-matrix interfaces in continuous fiber–reinforced ceramic matrix composites. The talks were organized into four sessions; mechanical modeling, test methods, interface behavior, and modification and control. The significance of the fiber-matrix interface in the design and performance of these materials is evident. Numerous mechanical models to relate interface properties to composite behavior, and interpret test methods and data, were enthusiastically discussed. Issues such as residual stresses, load transfer, fiber roughness, fiber coatings, and environmental stability were noted. Improved test methods and data analysis have provided more accurate and detailed information about the forces acting at this boundary, and enhanced the understanding of fiber debonding and sliding. A variety of new coating systems for improved performance and stability are being developed with some interesting new developments in interlayers for oxide-oxide composites.

One issue of great concern for any advanced material for use in extreme environments is stability. This theme arose frequently throughout the symposium and was the focus of discussion on the final day. Corrosion, thermal shock resistance, and cyclic thermomechanical loading were primary concerns for limiting the life of ceramic composites in many of the potential applications. Although great progress has been and continues to be made in the advancement and improvement of ceramic matrix composites, much more must be accomplished to fully understand these materials and optimize the properties of each system for specific applications.

The papers have been assembled into a proceedings that is to be published by the Materials Research Society.▲

CFCC News Wins Award

1994-95 Award for Distinction

“By winning the award, which is the highest given in each of the competition’s categories, CFCC News will be entered in STC’s international competition for publications.

CFCC News, the newsletter for the Continuous Fiber Ceramic Composite Program, was given the 1994-95 award for Distinction in Publications by the East Tennessee Chapter of the Society for Technical Communication (STC).

By winning the award, which is the highest given in each of the competition’s categories, *CFCC News* will be entered in STC’s international competition for publications. Of 113 entries in the East Tennessee competition, only *CFCC News* and the winners in five other categories will be submitted for the international judging.

CFCC News is prepared by Gloria Caton and Tim Elledge at Oak Ridge National Laboratory (ORNL), which is managed by Lockheed Marietta Energy Systems, Inc., for the U.S. Department of Energy. Merrill Smith, program manager for the CFCC Program, has overall responsibility for *CFCC News*.

In giving the top award to *CFCC News*, judges noted the newsletter’s

Continued on page 20

Materials, Science and Technology

Educators Meet

By Rick Lowden,
Oak Ridge National Laboratory

The 10th Annual National Educators Workshop will be held in Oak Ridge, Tennessee, November 6–8, 1995.

The "NEW: Update 95" workshop has been organized to provide current information on engineering materials technology to educators through plenary sessions, experiments, and other activities.

This year's theme is "Standard Experiments in Engineering Materials, Science and Technology" and CFCCs are again being highlighted in a combination of a mini–plenary session and laboratory demonstrations. Other topics include gel casting, microwave processing, materials joining, and wear and friction.

The Next Phase

The workshop marks the move into the next phase in the understanding of CFCCs. In a combination of lecture and laboratory experiments, the thermomechanical behavior and failure mechanisms of these materials will be examined.

Information about the testing and characterization of continuous fiber ceramic composites will be combined with the effects of exposure to service environments to demonstrate the various techniques and analyses used to qualify and understand ceramic matrix composites prior to acceptance for use in an application. The workshop will consist of three sections: thermomechanical testing, materials characterization, and environmental effects. All aspects of the workshop will be interconnected.

The goal of the workshop is to provide an overview of the methods and procedures used to develop engineering property data and detailed microstructural and compositional information about CFCCs that can be used in the selection of materials for specific applications and components.

In the previous mini-course, offered by Bill Long of B&W and Ken Reifsnider of Virginia Polytechnic Institute and University, the applications and benefits of CFCCs were introduced.

The program for the previous course also emphasized the design and use of and with these advanced materials for use in high-temperature industrial applications. Many of the basic issues concerning the "value" of these materials and their constituents, construction, and performance were discussed. That program was also presented to representatives from the Montana Science and Technology Consortium in a session prior to the Fall CFCC Working Group Meeting in Bozeman.

Sponsors

The NEW is sponsored by the U.S. Department of Energy, NASA Langley Research Center, NIST Materials Science and Engineering Laboratories, and the Schools of Technology and Science at Norfolk State University. James Jacobs of Norfolk State and Mike Karnitz of ORNL are co-chairing the workshop. Rick Lowden of ORNL is assisting with the session on CFCCs.

For more information concerning NEW '95, please contact:
Dr. James Jacobs, (804) 683-8109,
FAX: (804) 683-8215.▲

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"This year's workshop will move into the next phase in the understanding CFCCs. In a combination of lecture and laboratory experiments, the thermomechanical behavior and failure mechanisms of these materials will be examined.

CFCC NEWS

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

Managing Editor
Gloria M. Caton
E-mail: mgc@ornl.gov

Newsletter Designer and Technical Writer/Editor
Tim Elledge
E-mail: x3x@ornl.gov

Suggestions and contributions are invited and should be sent to:

Gloria M. Caton
Oak Ridge National Lab.
1060 Commerce Park
MS 6480
Oak Ridge, TN 37830

Phone: (423) 574-7759
Fax: (423) 574-9888
E-mail: mgc@ornl.gov

This newsletter is prepared at the request of the DOE Office of Industrial Technologies by the Health and Safety Research Division at Oak Ridge National Laboratory, which is managed by Lockheed Martin Energy Systems, Inc., for the U.S. Department of Energy, under Contract DE-AC05-84OR21400.

A COMMUNICATION OF THE CONTINUOUS FIBER CERAMIC COMPOSITE PROGRAM

Calendar

November 1995

Society of Engineering Science–32nd Annual Technical Meeting
October 29–November 1, 1995; New Orleans, Louisiana.
Call for papers.
Sponsor: Society of Engineering Science
Contact: David Hui, University of New Orleans, Mechanical Engineering, New Orleans, LA 70148. (504) 286-6192. Fax (504) 286-7413. E-mail: dxhme@uno.edu.

January 1996

20th Annual Cocoa Beach Conference & Exposition on Composites, Advanced Ceramics, Materials, and Structures.
January 7–11, 1996; Cocoa Beach, Florida.
Sponsor: American Ceramic Society, Engineering Ceramics Division.
Contact: American Ceramic Society Exposition Sale (614) 794-5844, Howard Johnson (407) 783-9222, Hilton (407) 799-0003.

Symposium on Thermal and Mechanical Test Methods and Behavior of CFCCs. Call for Papers.
January 8–9, 1996; Cocoa Beach, Florida.
Cosponsored by ASTM committees C28 “Advanced Ceramics,” E08 “Fatigue and Fracture,” and the American Ceramic Society's Engineering Ceramics Division. An ASTM Special Technical Publication of peer-reviewed papers resulting from the symposium is planned. Submit a title, 300-word abstract, and an ASTM paper submittal form by February 6, 1995 to Dorothy Savini, Symposia Operations, ASTM, 1916 Race St., Philadelphia, PA 19103-1187. (215) 299-5413.

CFCC News Wins 1995 Award

Continued from page 18

“consistently high standard” of “clear writing” as well as its “superb combination of color, graphics, and eye-catching devices,” which “help to make the technical subject interesting.”

The judges also cited “innovative” design elements as part of a format that “invited one to keep reading.”

STC’s South Carolina chapter judged this year’s competition, which in addition to newsletters included categories such as annual reports, informational brochures, and technical reports. STC is the world’s largest professional organization for technical publications, with more than 14,000 members in over 120 chapters.▲