CFCC News

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Milestone in using SiC/SiC CFCC liners in gas turbines

By Narendernath Miriyala and Jeffrey Price, Solar Turbines, Inc.

In the current field test, which began in April 1999, SiC/SiC liners are being protected for the first time with environmental barrier coatings.

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Yet another milestone in the CSGT program was reached when an 8000-h test of a Centaur 50S engine retrofitted with EBC-protected SiC/SiC liners began in the second half of August 1999 at Malden Mills Industries in Lawrence, Massachusetts.

> "The engine selected for ceramic component insertion under the CSGT program is Solar's Centaur 50S engine, which has a rated shaft thermal efficiency of 29.6% and an electrical output of 4.1 MWe.

DOE-initiated program

The U.S. Department of Energy (DOE), Office of Industrial Technologies (OIT), initiated the CSGT program to demonstrate ceramic technology by selective replacement of cooled metallic hot-section components with ceramic parts (blades, nozzles, and combustor liners) in an industrial gas turbine for cogeneration operation.

The CSGT program, with Solar as the prime contractor, started in September 1992. Currently, the program is in the field-engine-testing phase (Phase III). The major contributors to the successful development and testing of CFCC liners are

1. AlliedSignal Composites Incorporated (ACI)
2. BFGoodrich Aerospace (BFG)
3. Oak Ridge National Laboratory (ORNL)
4. Argonne National Laboratory (ANL)
5. Pratt & Whitney (P&W)
6. United Technology Research Center (UTRC)
7. Texaco

Solar's Centaur 50S engine

The engine selected for ceramic component insertion under the CSGT program is Solar's Centaur 50S engine, which has a rated shaft thermal efficiency of 29.6% and an electrical output of 4.1 MWe.

The engine operates at a turbine-rotor-inlet-temperature (TRIT) of 1010°C. A SoLoNO$_x$ combustion system is used in the all-metal engine to obtain NO$_x$ emissions of < 25 ppmv and CO emissions of < 50 ppmv. Under the CSGT program, the engine hot section (Fig. 2) was redesigned to effectively retrofit the two metallic cylinders (inner liner and outer liner) in the annular-combustor with CFCC parts.

The two liners form the primary combustor zone and operate under the most severe operating conditions, including high flame temperatures and direct flame impingement. The outer surface of the inner liner and the inner surface of the outer liner form the working surfaces in the combustor.

Composite system chosen

Based on a series of subscale combustor rig tests in Phase II of the CSGT program, a SiC/SiC composite system was chosen over monolithic SiC for field-engine tests. In the current design, the nominal dimensions of the inner liner are 33 cm inner diameter,

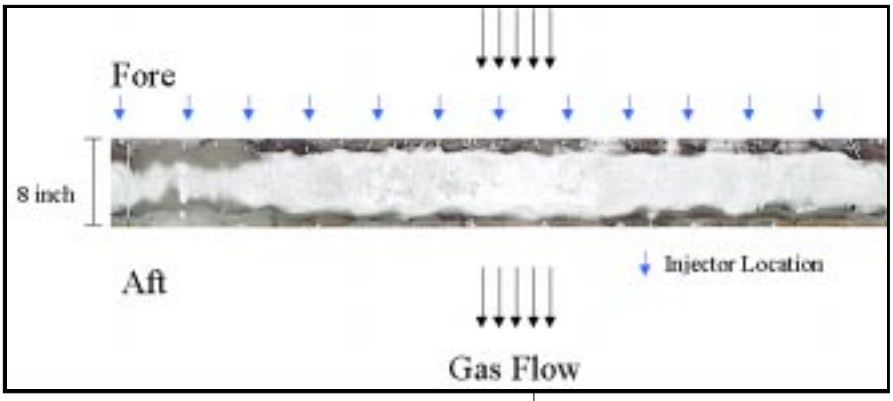

Fig. 3. Inner surface of the Hi-Nicalon/Enhanced-SiC outer liner after 5016 h field-engine testing in CSGT.

thin SiC seal coat) were fabricated by ACI using the chemical vapor infiltration (CVI) technique.

This test was terminated after 948 h of full-load engine operation. Before installation at the test site, the engine was subjected to a 100-h acceptance test at Solar. Thus, the CG-Nicalon/Enhanced-SiC liners were exposed to engine conditions for a total of 1048 h. After this exposure, the CFCC liners were heavily oxidized, particularly the inner liner.

Second Test

A second field-engine test of CFCC liners began in March 1998. For this test, several design and material changes were incorporated in the combustor system to increase the durability of the CFCC liners. Design changes to improve the conduction path from the CFCC liners to the metallic housings resulted in reduced hot-wall temperatures.

Material changes to the CFCC liners included the substitution of CG Nicalon fibers with Hi-Nicalon fibers, increased material density, and increased seal coat thickness. After 2114 h of field-engine testing, "white glass" formation was observed on the outer surface of the inner liner. At this point, it was decided to stop the test at the earliest opportunity, so that the recession of the seal coat in the engine environment could be correlated to that observed in a simulated combustion environment furnace at ORNL (15% steam +85% air, 10 atmospheres pressure, 1204°C).

Consequently, the field test was stopped after the Hi-Nicalon/SiC liners were exposed to engine conditions for 2258 h. The inner liner was sectioned for detailed microstructural examination. It was observed that the degradation of Hi-Nicalon/Enhanced-SiC in the CSGT engine closely matched the degradation in the ORNL furnace.

Low-cost test bed

Based on this finding, the simulated combustion environment furnace is now extensively used as a low-cost test bed to screen various ceramic material systems/coatings.

The Hi-Nicalon/Enhanced-SiC outer liner used in the second field-engine test was reused in the third field test, which started in December 1998. The new inner liner used in this test was a Hi-Nicalon/SiC component fabricated by BFG using the melt infiltration (MI) technique.

The field test was concluded after 2758 h of full-load engine operation when the outer liner surpassed more than 5000 h of exposure to combustion environment. The inner surface of the outer liner after 5016 h of field-engine testing is depicted in Fig. 3.

Composite behavior was observed on the fracture surfaces of specimens obtained from the two liners, even from areas where severe oxidation occurred on the outer liner.

The oxidation was limited to the surface of the MI inner liner. Both surface oxidation and internal degradation were observed in the CVI outer liner. A similar behavior was also observed in the ORNL furnace. Ther-

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Infrared burners

Employing rare-earth garnet fibers for paper industry

By R. W. Goettler and R. A. Wagner, McDermott Technology, Inc.

McDermott Technology, Inc. (MTI) is developing chopped, or discontinuous, rare-earth garnet fibers for a variety of applications.

The key near-term need for these fibers is for use as the matrix phase of porous CFCC gas-fired infrared (IR) burners for the paper industry. These burners are used to dry paper in the paper-making process and to dry coatings applied to the paper.

The reason for utilizing an oxide-based CFCC IR burner is that the radiation spectrum that is transmitted to the paper by the burner can be controlled to optimize the drying process.

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Current drying methods include steam boxes, black body IR burners, and electric IR heaters. Each of these methods has limitations that are addressed by the CFCC IR emitter. While the steam box transfers a considerable amount of energy to the paper web, very little penetration is achieved and a highly nonuniform temperature profile is produced in the web.

Other heaters

Conventional gas-fired IR heaters produce black body radiation that also exhibits little penetration in the paper web. Finally, electric IR heaters produce very short wave length radiation that penetrates through the web, with little heat transfer to the web.

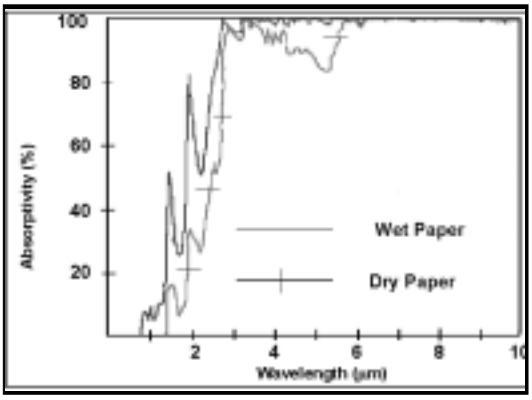

Fig. 1. Absorptivity of wet and dry paper web.

The MTI CFCC emitter is designed to produce a controlled radiation spectrum that will optimize heat transfer to the paper web and produce a uniform temperature profile. The radiation spectrum is controlled by the addition of rare-earth elements to the composite structure. The selective emission spectrums of the rare-earth elements result from inner electron shell transitions.

The emission peaks in the spectrums for erbium and holmium match the water absorption wavelengths.

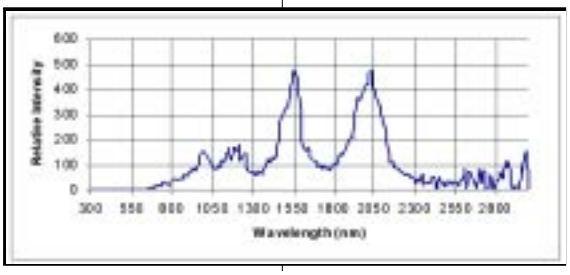

Fig. 2. Spectral emittance of Er/Ho oxide mixture

Figure 1 shows the absorption spectrums for dry and wet paper. The peaks at 1500 and 2000 nm match the peak emission wavelengths for erbium and holmium, respectively.

Chopped fiber

MTI has chosen to develop a chopped fiber with the composition $(Er_{0.5}, Ho_{0.5})_3 Al_5O_{12}$, also termed ErHoAG, for use in the CFCC IR burners. The spectrum for an Er/Ho oxide mixture is shown in Fig. 2. The potential advantages of a selective emitting burner are

1. lower fuel consumption;
2. faster heat-up and cool-down cycles, helping to prevent fires during line stoppages; and
3. faster throughput of product.

MTI has developed proprietary precursor chemistries for rare-earth garnet fibers. These water-based metal-salt precursors have excellent spinning characteristics.

The precursors are being investigated by 3M for use in making continuous yttrium aluminum garnet (YAG) fibers for potential use by MTI in oxide-oxide CFCC turbine combustor liners. MTI is concentrating on developing the processes for making the chopped ErHoAG fibers in suitable quantities for IR burner demonstration. MTI uses a 3.5-in-diam fiber spinner that is a thin-walled container with multiple holes along the outside wall through which the fiber is extruded and drawn while spinning at up to 7500 rpm.

High-pressure air jets are positioned to direct the spun/drawn fiber to a collection site. Figure 3 shows fiber being spun at MTI. The SEM (Scanning Electron Microscope) photomicrograph of Fig. 4 shows some garnet fibers produced by MTI.

Avoiding flaws

The green fibers generally have an oxide yield of about 40 weight percent. To produce a high quality fiber, it is important that the pyrolysis schedule be carefully controlled to avoid the generation of strength-limiting flaws that could occur as volatile species are evolved.

Fig. 3. Spinning of chopped ErHoAG fiber at MTI.

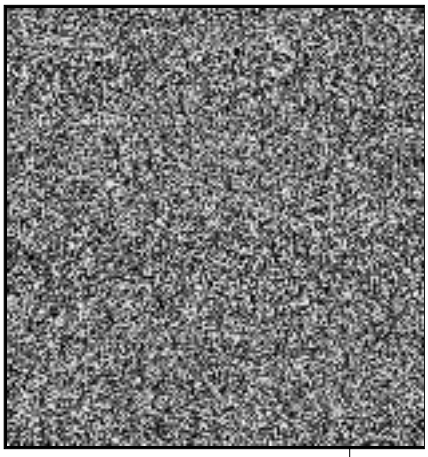

Fig. 4. SEM Photomicrograph of spun garnet fiber.

The heating rate through both endothermic and exothermic events during the pyrolysis cycle is important for assuring uniform densification of the fiber during the firing cycles. MTI is in the process of optimizing both the pyrolysis and firing schedules for the ErHoAG chopped fiber to achieve a high-strength fiber.

Although emissivity properties are the most important for this application, moderate- to high-strength fibers are required to produce a porous CFCC body with the greatest durability possible. In addition, fiber compositions that show some grain stability for prolonged exposure to the high operating temperatures (1200° to 1300°C) are required for strength retention purposes. The garnet composition is known to exhibit such

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Protecting CFCCs

Environmental Barrier Coatings

> "Environmental barrier coatings will protect the underlying silica-forming ceramic from rapid degradation in high-pressure, high-velocity, moisture-rich combustion environments.

By J. A. Haynes,
Oak Ridge National Laboratory

Recent laboratory and engine tests of Si-based ceramics have revealed that protective coatings will be necessary to ensure the long-term environmental stability of CFCC components in combustion environments.

The high-temperature oxidation resistance of CFCC materials is contingent on the formation of a silica (SiO_2) scale that serves as a slow-growing diffusion barrier and provides excellent protection in dry oxidizing conditions.

However, actual combustion environments generate significant amounts of water vapor (typically at elevated pressures) and impurities, which can rapidly degrade protective silica scales by a variety of mechanisms. These include: (1) acceleration of silica growth rates by water vapor and alkali impurities, (2) volatilization and/or erosion of silica in the presence of water vapor and high-velocity combustion gases, and (3) hot corrosion by reaction with molten alkali salt deposits.

Based on these corrosion-related performance limitations, CFCC components will require protective coatings that reduce or eliminate silica degradation.

Barrier coatings

Environmental barrier coatings (EBCs) will protect the underlying silica-forming ceramic from rapid degradation in high-pressure, high-velocity, moisture-rich combustion environments.

Candidate EBC materials must exhibit: (1) good high temperature phase stability, (2) chemical compatibility with SiC, (3) good adhesion, (4) thermomechanical stability on SiC-based materials, (5) resistance to degradation by water vapor, (6) good

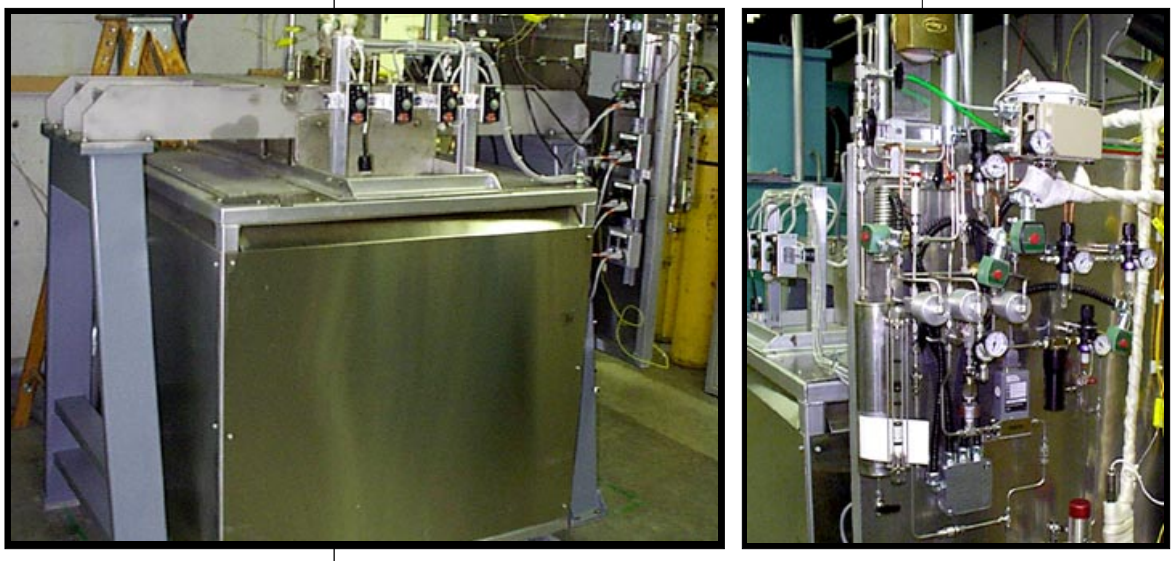

Fig. 1. ORNL high-pressure, isothermal steam rig and gas flow assembly.

Fig. 2. ORNL automated cyclic oxidation test rig.

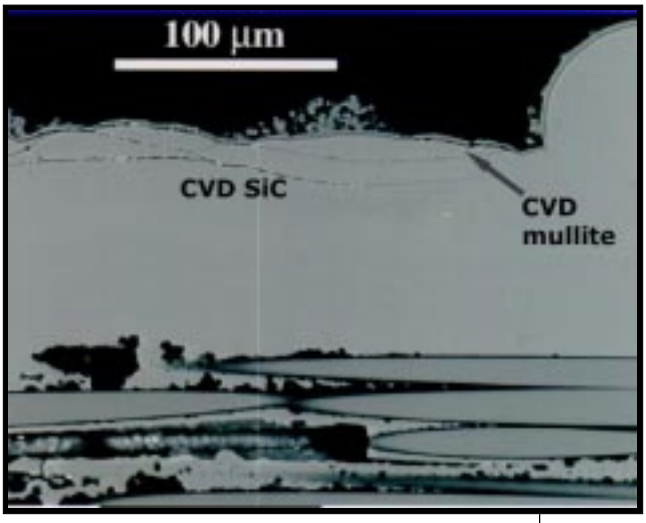

Fig. 3. CVD mullite coating on a CFCC substrate with a CVD SiC seal coat after 500 h exposure at 1204°C and 10 atm in air + 15 vol% H_2O.

hot corrosion resistance, and (7) cost-effective fabrication. It is likely that most EBCs will consist of multiple coating layers in order to meet these diverse requirements.

Development of EBCs

Oak Ridge National Laboratory (ORNL) is testing a wide variety of candidate EBC components or systems in simulated combustion environments in order to assess the current state of the art in EBC technology.

At present there is little data on the high-temperature corrosion behavior or thermomechanical durability of ceramic coating materials in high-pressure water vapor. To address this issue, ORNL has built two types of corrosion rigs specifically designed for testing in moisture-rich environments.

One type of rig, shown in Fig. 1, allows isothermal testing of multiple specimens at elevated pressures and controlled moisture contents. Typical test conditions are 1200°C, 10 atm total pressure, and 15 vol% water vapor in air (300 cm^3/min) for time periods ranging from 100 to 500 h.

Microstructural comparisons of silica-forming materials tested in a gas turbine engine and the ORNL high-pressure steam rig confirmed similar rates and modes of silica formation. It was concluded that the ORNL high-pressure steam rig is a viable and cost-effective method of screening candidate EBC materials.

Variety of materials tested

A variety of Si-based materials have been tested in order to obtain preliminary information on silica growth rates, volatility, and microstructures as a function of pressure, temperature, and moisture content.

Candidate EBC materials are being tested under the same conditions to evaluate their effectiveness in reducing silica growth rates. A number of candidate materials and single- and multilayer coatings on CFCC substrates are being tested, including mullite and alumina-based coatings, mullite/YSZ, materials from the NZP

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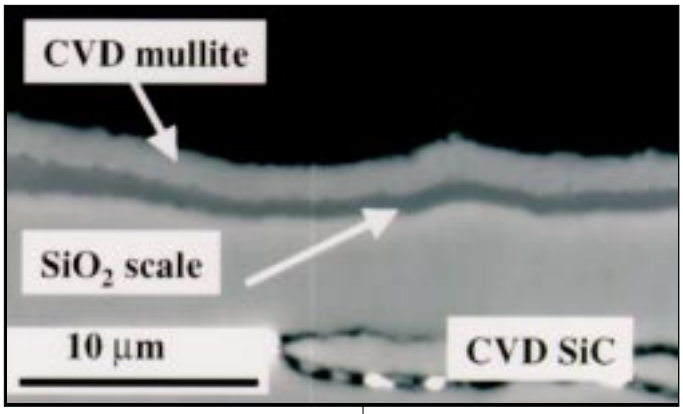

Fig. 4. CVD mullite on CVD SiC after 500 h exposure at 1204°C and 10 atm in air + 15 vol% H_2O. Note the very thin silica scale.

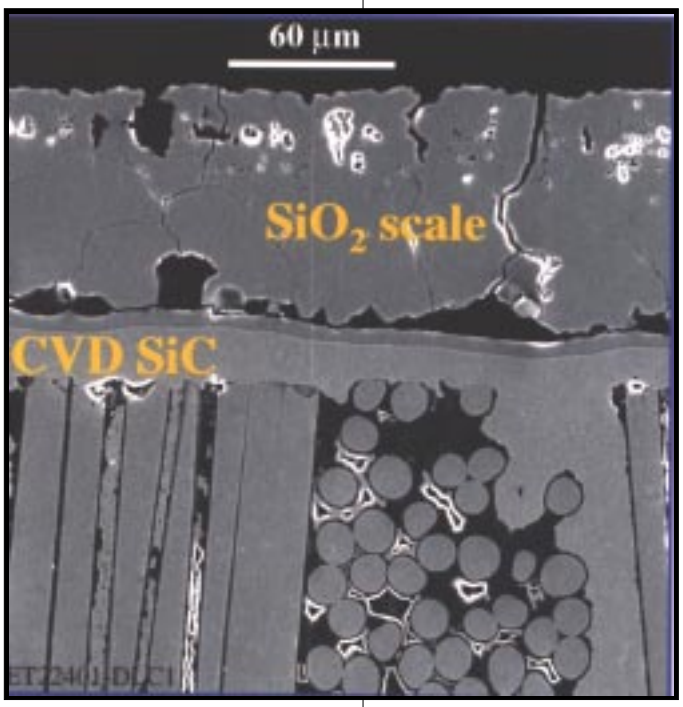

Fig. 5. Non-coated CVD SiC after 500 h exposure at 1204°C and 10 atm in air + 15 vol% H_2O. Note the very thick defective silica scale.

(Na-Zr-P) family, as well as a number of proprietary coating systems.

Automated rig

The second type of test rig (Fig. 2, previous page) is automated in order to allow rapid thermal cycling of multiple specimens in ambient pressure atmospheres containing controlled amounts of water vapor (1-h cycles to 1200°C in air + 50% or 90% water vapor). These cyclic tests provide a means of assessing the combined effects of water vapor and thermal stresses on the adhesion and oxidation behavior of various ceramic coating systems.

An additional EBC-related task addresses fabrication and testing of protective mullite coatings for operation in moisture-rich environments. Mullite ($3Al_2O_3 \cdot 2SiO_2$) is a prime candidate for EBC systems, due to its good high-temperature strength, corrosion resistance, and excellent thermal expansion match with SiC.

Plasma-sprayed mullite coatings for Si-based ceramics have been developed at NASA Glenn Research Center. However, disadvantages of plasma-sprayed mullite in moist environments include inherent porosity and microcracking, residual amorphous material, and relatively high levels of alkali impurities. There are also indications that plasma-sprayed mullite experiences preferential volatilization of silica when water vapor is present.

Chemical vapor deposition

Crystalline mullite coatings can also be deposited by chemical vapor deposition (CVD), as first demonstrated at Boston University. Advantages of fabrication by CVD include the capability of depositing dense, high-purity coatings.

Various types of CVD mullite coatings have been deposited onto CFCC substrates with CVD SiC seal coats (supplied by Allied Signal Composites) at ORNL.

Isothermal testing in high-pressure water vapor revealed that dense CVD mullite coatings (Fig. 3) are capable of dramatically reducing the oxidation rates of CVD SiC seal coats. Specimens with thin CVD mullite coatings (2–4 µm) formed 0.7–1.5 µm of dense, amorphous silica after a 500-h exposure (Fig. 4).

No microstructural evidence of mullite volatility was observed under the low flow rate conditions of these steam rig tests. By comparison, noncoated CVD SiC formed highly defective silica scales of up to 130 mm in thickness (Fig. 5) after an identical 500-h steam rig test. Current efforts are focused on developing CVD mullite coatings with optimized microstructures and Al/Si ratios for EBC applications.Δ

■ AlliedSignal & DuPont Lanxide Composites

Just over a year ago, AlliedSignal announced the acquisition of DuPont Lanxide Composites (DLC) in Newark, Delaware.

AlliedSignal purchased 100% of the outstanding shares of stock from both DuPont and Lanxide (the previous owners of the joint venture) and renamed the corporation AlliedSignal Composites Incorporated or ACI.

Stan Hemstad of AlliedSignal Composites provided the following details about what this purchase might mean for the CFCC industry.

The acquisition includes ceramic matrix composites produced by chemical vapor deposition, several Directed Metal Oxidation (DiMOX) processes and materials patents, Ceraset Pre-ceramic polymer, and PRD-66 glass fiber-reinforced Ceramic Matrix Composites.

The acquisition also provided for the extension of the Lanxide held technology, Ceraset, and DiMOX to friction applications.

Promising applications include PRD-66 candle filters for removing ash from coal combustion products in the emerging clean coal combustion power generating technologies and hot section parts for aircraft turbine engines, including the combustion liner being developed for the High Speed Civil Transport under the NASA Enabling Propulsion Materials Technologies program.

Other products in development include recuperators for high-temperature heat transfer, radiant burners for many industries, and hydrogen turbopumps and exhaust nozzles for reusable launch vehicles.

ACI is part of AlliedSignal's Aircraft Landing Systems, one of four strategic business units that belong to AlliedSignal's Aerospace Equipment Systems (AES). ACI has many potential internal customers including AlliedSignal Engines, already a customer; Environmental Control Systems, a manufacturer of heat exchangers used in the aerospace industry; Engine Systems and Accessories, a supplier of control systems and hardware for missiles, launch vehicles, and aircraft/power turbines; and the newly formed AlliedSignal Power Systems that is entering the market as a supplier of small power-generation turbine engines.

The ceramic composite technologies in AlliedSignal Composites Inc. are a strong complement to AlliedSignal's Ceramic Components Division, which manufactures in-situ toughened silicon nitride for the monolithic ceramic market. For information, contact Stan Hemstad, E-Mail: Stan.Hemstad2@alliedsignal.com. Telephone: 302/456-6586.

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CFCC News Update

Measuring Properties

Acoustic techniques applied

By Bill Ellingson and J.G. Sun, Argonne National Laboratory, and T.A.K. Pillai, University of Wisconsin

As part of the Nondestructive Characterization (NDC) project, Argonne National Laboratory (ANL) has been working to develop faster, lower cost methods for characterizing CFCC materials.

One common relatively low cost NDC (often also referred to as NDE) method is C-scan ultrasound.

Usually C-scan ultrasound involves placing the test component into a water bath to allow the ultrasound to be coupled to the component. Fig. 1 shows a typical C-scan ultrasound set up. By scanning the component under study, a 2-D map of the materials acoustic attenuation is displayed.

With many CFCC materials, especially those using Chemical Vapor Infiltration (CVI) as the infiltration method or Polymer Impregnation and Pyrolysis (PIP) as the infiltration method, submersing the component into water is not desirable because of possible contamination.

ANL in collaboration with QMI in Costa Mesa, California, has demonstrated the use of a new technology, air-coupled ultrasound (ACUT), for characterizing CFCC materials.

Figure 2 shows a schematic diagram of an ACUT C-scan system. In this system, the component is tested in ambient air, and no water or other coupling medium is necessary.

Obviously this eliminates any contamination other than that present in the laboratory or test environment.

The most critical component of an ACUT system is a pair of well-

> *"In this system, the component is tested in ambient air and no water or other coupling medium is necessary.*

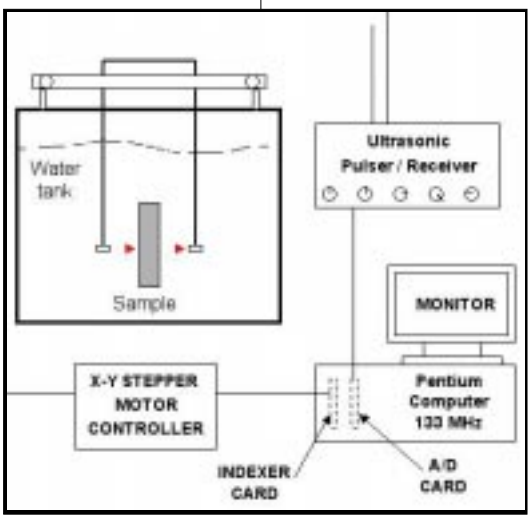


Fig. 1. Schematic diagram of typical C-scan ultrasonic system.

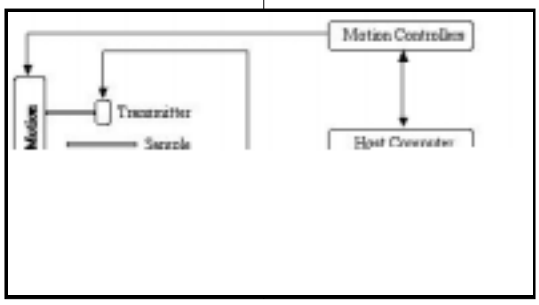
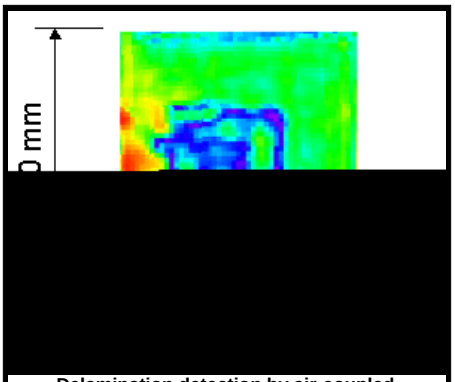
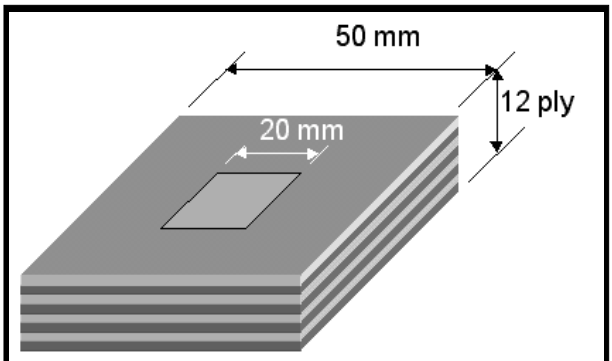


Fig. 2. Schematic diagram of air-coupled ultrasound C-scan system.



Delamination detection by air-coupled C-scan.



CVI SiC/SiC (uncoated) CG-Nicalon 40% fiber vol. (plain weave) 0.3 um pc interface layer. Simulated defect between layers 8 and 9 500 um thick nominal 10% porosity.

Fig. 3. Schematic diagram and resulting air-coupled ultrasonic C-scan of SiC/SiC CVI CFCC specimen.

designed efficient transducers. Although there are several ways of generating and receiving ultrasound waves in air, two methods have received most technological attention. The first uses a capacitance device which employs a flexible membrane that oscillates between two precision machined electrodes.

Sending/receiving device

For the transducer to act as a sending device, a voltage is applied to the electrodes, and for the device to act as a receiver, energy received by the membrane causes a change in an applied bias voltage. The advantage of these devices is that they have a relatively broad band width allowing for digital signal processing. The disadvantage is that the signal to noise (S/N) ratio tends to be low.

The second method, the one used for our work, employs a classical piezoelectric crystal (PZT). The difference between a conventional UT transducer and the ACUT transducer primarily lies in the special acoustic matching lens placed on the face of the transducer.

In the work here, the matched transducers operate at a frequency of 400 KHz and have a focal length of 3.8 cm. In classical ultrasound received signal methodology, the received pulse is "gated." That is, only a selected portion of the received signal is, through the use of digital signal processing, allowed to be used as the signal representing the type of feature to be observed.

Received signal

Figure 3 shows a simple flat plate test specimen made with an intentional simulated delamination in the center and the resulting C-scan "image" map. Clearly the delamination is detected by simple observation. The scanning speed is very high, 7.6 cm/s.

Thus when properly set up, the system can operate totally unattended and will acquire data at very high rates. Thus, inherently, it is an inexpensive technology to implement.

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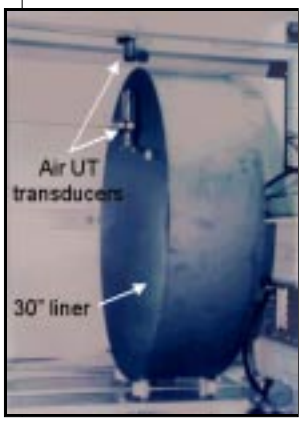

Fig. 4. Photograph showing the 30-in. CVI SiC/SiC CFCC combustor liner in the air-coupled ultrasound system.

One disadvantage of the PZT transducer is the very narrow band width. This prevents extensive digital signal processing, which can often be used to extract significant information about the material under study. The big advantage in using the PZT transducers is the high S/N ratios achievable.

Turbine Combustor Liners

One recent application of the use of the ACUT system has been to examine large full-scale SiC/SiC combustor liners under development for use in the Ceramics In Stationary Gas Turbine (CSGT) program, which is part of the U.S. DOE program for Advanced Turbine Systems (ATS).

To accommodate these large 30-in-diam. liners, a special rotation mechanism was designed, fabricated and tested. Figure 4, on page 11, shows the ACUT system with the 30-in. liners in place. By rotating the liner and translating the transducers back and forth, a 2-D "image" or C-scan of the entire liner can be obtained.

Figure 5 shows the resulting 2-D "image" data. What now is critical, of course, is the interpretation of the data. While an extensive data base is now being developed, test data obtained to date have been either corroborated by using one other of our NDC methods, namely thermal imaging or x-ray computed tomography or by destructive analysis.

Figure 6 shows the correlation between air-coupled ultrasound data, thermal diffusivity image data, and destructive analysis for an oxide/oxide 20-cm-diam. test liner. Note that the air-coupled UT data corresponds to destructive verification of a delamination.

Processing parameters

One very different outcome of using ACUT as opposed to water-coupled UT can be seen in Fig. 7. Figure 7 shows how thermal diffusivity increases in PIP SiC/SiC materials as a function of density and also shows the strength of the through-transmitted ultrasound signal as a function of density (porosity).

Normally, one would expect to see an increase in the strength of the transmitted signal as density increases. For air-coupled ultrasound in CFCC materials, it is just the opposite. The reason can be explained by the change in acoustic impedance as the CFCC materials become more dense.

Another application of the use of air-coupled ultrasound has been to study the extent of impact damage in CFCC panels. A set of PIP-processed SiC/SiC panels was impacted using a pendulum with different impact energies.

By scanning the samples with air-coupled ultrasound, as well as deter-

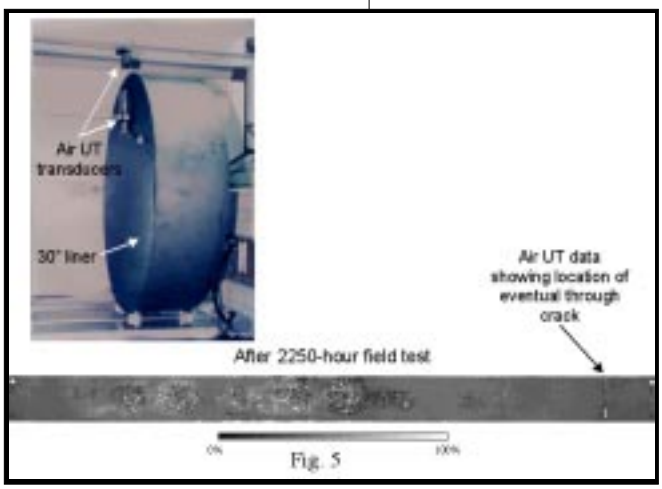

Fig. 5. Air-coupled ultrasonic C-scan data for 30-in. CVI CFCC combustor liner used for the CSGT project as part of the ATS program.

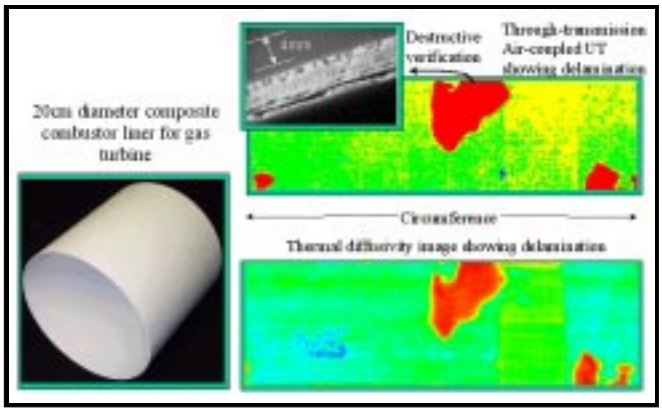

Fig. 6. Correlation between destructive analysis and ACUT and thermal image data.

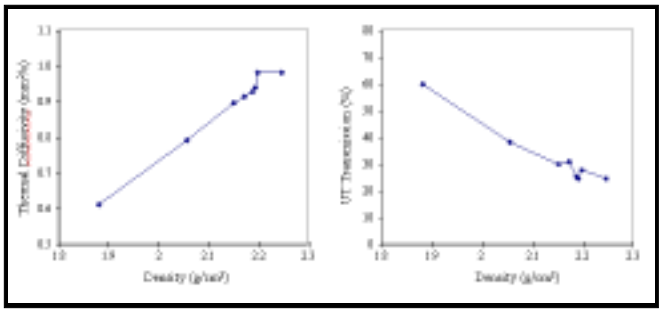

Fig. 7. Effect of CFCC density on thermal diffusivity and through transmitted ACUT signal.

mining the thermal diffusivity mapping, one can determine the area of the effected regions. Figure 8 shows a typical air UT scan along with a thermal diffusivity scan for one impacted panel. By determining the area of the effected region, and knowing the retained strength, one can obtain a relationship between NDC determined area and retained strength. This relationship is shown in Fig. 8.

Clearly, if a large enough data base for such conditions could be obtained, then critical decisions about the extent of impact damage could be determined entirely nondestructively.

One issue in many such situations is the need for one-sided access. Indeed one-sided access technology is now being developed. Figure 9 shows such an arrangement. In this case, both transducers are on the same side and the scan requires that only one side of the component be accessible. All other parameters remain the same.

Melt-Infiltrated CFCCs

Recently, with the development of Melt Infiltrated (MI) CFCC materials, the use of standard water-coupled ultrasound (WCUT) has again become useful.

MI materials often may be immersed in water. Recently Argonne has installed a water coupled ultrasound system. The system was provided to ANL by Deere and Company. The sensitivity of the use of WCUT system was recently demonstrated to detect an upset in the processing of a combustor liner for the ATS program.

Figure 10 shows a photograph of a 13-in.-diam. liner and the corresponding water-coupled and air-coupled ultrasonic "images" for the entire surface.

Destructive examination of the liner indicated that a large change in porosity resulted from the processing upset. It is important to note that the WCUT and ACUT reliably detected the change in porosity. It is clear that use of such technology in the processing stages could save valuable time and hence reduce component costs.

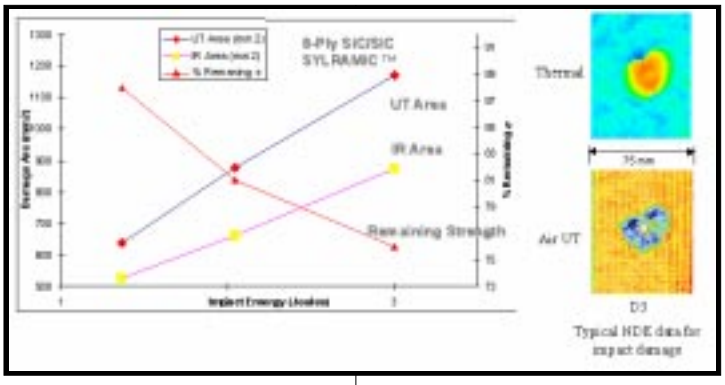

Fig. 8. Relationship between NDC-determined size of impact damage zone and retained strength.

Conclusions

As part of the NDC development project, air-coupled ultrasound has been demonstrated to provide a fast system for detection of changes in porosity, delaminations, and possibly impact damage. Further, if used in the one-sided access mode, the potential exists for using the technology in-situ, reducing the necessity to remove components from service.

Correlations between the ACUT data and thermal diffusivity NDC data have been demonstrated to have correlations with destructive analysis. Recently, for MI-processed CFCC materials, the use of water-coupled ultrasound has been demonstrated to be effective in detecting porosity.Δ

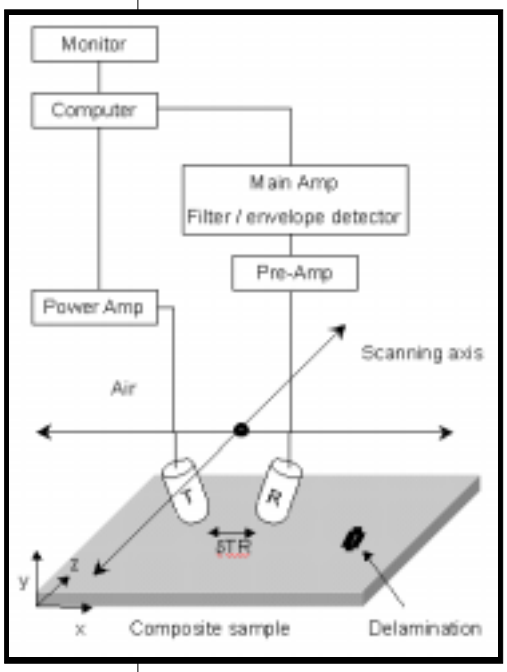

Fig. 9. Schematic diagram showing one-sided arrangement for air-coupled ultrasound.

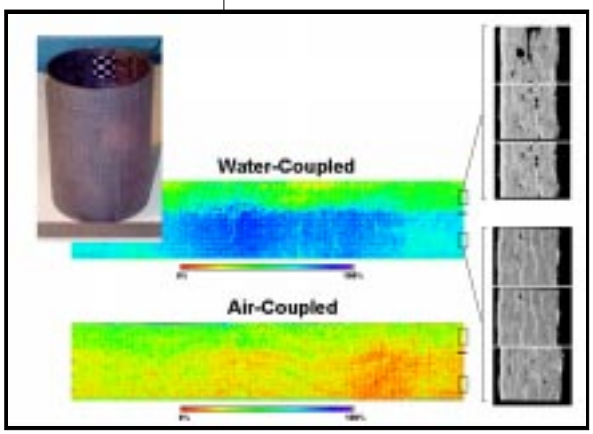

Fig. 10. Photograph and ultrasound "image" data for a 13-in. melt infiltrated combustor liner.

Into the (not so) distant future

Opportunities for advanced ceramics

> "Key material challenges for replacement materials include wear, corrosion, degradation at elevated temperatures, creep, mechanical and thermal fatigue, and impact and thermal shock.

By Douglas Freitag, Bayside Materials Technology, and David Richerson, Richerson and Associates

We conducted a study to assess the potential of advanced ceramic-based materials to help the Industries of the Future (IOF) meet their long-range visions of energy efficiency and pollution reduction, improved capital effectiveness, and increased productivity.

Industries considered include aluminum, chemicals, forest products, glass, metals casting, and steel.

Three objectives

The study had three objectives:
(1) provide to IOF a review of the status and potential of key advanced ceramic-based materials, including examples of specific application challenges these materials already are solving in other industries;
(2) provide to ceramic materials suppliers a review of key materials needs in each Industry; and
(3) provide recommendations of specific ceramic material candidates for specific applications in each Industry.

A variety of metal alloys and refractory ceramics being used by IOF were identified as candidates for replacement with advanced ceramic materials. While these conventional materials have gained widespread acceptance, their continued use is a source of high maintenance, lost productivity, and low energy efficiency.

Key material challenges for replacement materials include wear, corrosion, degradation at elevated temperatures, creep, mechanical and thermal fatigue, and impact and thermal shock.

Ceramics have superior wear resistance, corrosion resistance, and high-temperature stability, but they are often not selected for IOF applications because of higher cost and reliability concerns associated with mechanical stresses or thermal shock.

The study determined that while major improvements have been achieved in ceramic materials in recent years in their mechanical and thermal properties and in design knowledge, costs remain high and user confidence remains low. In many cases, lack of awareness on the part of the IOFs prevents advanced ceramics from even being considered.

Four categories of ceramics offer benefits: monolithics, coatings, refrac-

Table 1. Summary of applications identified for advanced ceramics within the Industries of the Future

Aluminum
- heat exchanger tubes
- burners
- refractories
- wettable cathodes
- inert anodes
- scrubbers
- wear parts
- corrosion resistant parts
- thermocouple sheaths
- immersion heaters

Forest Products
- suction box covers
- foils and guides
- paper cutters
- bearings
- pump valves & seals
- rolls
- smelt spouts
- heat exchanger tubes
- radiant burners
- hot gas filters
- sensors

Chemicals
- catalyst supports
- igniters
- spray nozzles
- heat exchanger tubes
- heat exchanger headers
- recuperators
- reformer tubes
- refractories
- fans
- valve seats, inserts, and bodies
- sensor shields
- pump impellers
- knives
- molds
- pump housings
- filters
- membranes

Metal Casting
- crucibles
- molds, cores, and dies
- nozzles
- valves
- tap hole linings
- thermocouple sheaths
- molten metal pumps
- support tubes—induction heaters
- burners
- refractories

Refining
- reformer tubes
- particle separators
- fans
- tube hangers
- thermal sleeves
- valves
- heat exchanger tubes

Glass
- thermocouple sheaths
- refractory insulation
- molds and dies
- fans
- bubblers
- lances
- forming lips
- tweels
- burners
- rolls

Steel
- burners
- cutting tools
- fans
- coke injection tubes
- tap hole nozzles
- sensor shields
- electrodes
-

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This study identified a variety of metal alloys and refractory ceramics that are currently being used by industry that are candidates for replacement with advanced ceramic materials.

Into the future

(Continued from page 15)

material needs of the IOFs. Table 1 lists a representative cross-section of these opportunities. As part of the study, recommendations were developed to provide a meaningful and achievable path by which advanced ceramics can be used more often.

Education/awareness
There is room for improvement in communication to IOFs on advances in ceramic materials and to the advanced ceramics industry on the IOF's needs and challenges. Recommendations:

1. Disseminate the results of the study through direct mailing to IOF partners and advanced ceramic companies and, more widely, through the World Wide Web (WWW).

2. Increase knowledge by the advanced ceramics industry on the vision, roadmaps, projects, and solicitations for funding currently being addressed under each of the Vision Industries. The informative Web site of the Department of Energy's Office of Industrial Technology (OIT), provides much of this information, including the OIT Resource Catalog from which over 450 OIT publications can be ordered or viewed in HTML format.

3. Increase participation on the part of the advanced ceramics industry at IOF conferences and expositions both in the form of displays and presentations on the status of advanced ceramic materials and their past and potential uses in the respective IOFs.

4. Establish a Web site that provides easy access to information on advanced ceramics for use by the IOFs. Much of this information exists within various sources on the Internet, but a centralized source with links and expanded information on specific problems would be a valuable communication resource. In addition, data that exist throughout the advanced ceramics industry but not currently on the Internet should be made available on this site.

5. Develop new and creative ways to communicate information, including articles in industry trade publications, WWW sites, papers and lectures at trade gatherings, and video and CD ROM reports.

6. Arrange tours of IOF plants by the advanced ceramics industry. The goal of these tours is to see first hand what the problems are and furnish immediate feedback as to how advanced ceramics could provide a potential solution.

> *"The study identifies opportunities that could benefit from patrnerships.*

Partnership facilitation
The study identifies opportunities that could benefit from partnerships. Recommendations:

1. Encourage interaction and formation of partnerships among the advanced ceramics community and IOFs. Many opportunities identified in this study are considered precompetitive to specific industry groups and are of sufficient complexity that a consortium or partnership between the specific IOF and advanced ceramics industry will be required to develop timely solutions.

2. Review the results and outcomes from the recent joint meetings of the Chemical and Glass Industries with the advanced ceramics industry.

Challenges
Another conclusion of the study is that a number of crosscutting technical challenges remain before the

> *"As part of the study, recommendations were developed to provide a meaningful and achievable path by which advanced ceramics can be used more often.*

widespread application of advanced ceramic materials will occur in the 1OFs. Some of these challenges include reduced cost, improved reliability, scale-up, increased design knowledge, and expanded design databases. Recommendations:

1. Support crosscutting programs like the CFCC Program that are important in ensuring that early-stage research efforts support energy-intensive IOFs.

2. Do regular reviews of remaining crosscutting barriers to the insertion of advanced ceramics. Consider using new crosscutting research and development programs for the demonstration of key technology platforms for improving energy efficiency and reducing environmental problems.

3. Improve databases, test methods, and design guidelines for use by the IOFs through increased coordination with ASTM, ASME, Mil-Hdbk-17, and other related activities. Current databases are generally inadequate and do not allow IOF experts to make favorable design decisions on advanced ceramics. Test methods are frequently lacking, and design guidelines are not well understood when applied to advanced ceramic materials.

Partnership gets results

Through the continued partnership of DOE and the advanced ceramics industry, many of the recommendations are beginning to produce results.

Over 2,000 copies of the report have been distributed to industry. The CFCC segment of the advanced ceramics industry has targeted and participated in conferences sponsored by the National Association of Corrosion Engineers and American Society of Mechanical Engineers.

Opportunities for partnerships between the advanced ceramics industry and IOFs continue to grow with joint meetings planned with the aluminum and forest product industries and plant tours being discussed with the glass industry.Δ

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Through the continued partnership of DOE and the advanced ceramics industry, many of the recommendations are beginning to produce results.

The full study was published as:

DOE/ORO 2076 Opportunities for Advanced Ceramics to Meet the Needs of the Industries of the Future, December 1998, by Douglas Freitag and David Richerson.

It is available from:

DOE-OIT Resource Room, 5F064, EE-20, 1000 Independence Ave., SW, Washington, DC 20585.

Contacts:

Marilyn Burgess or Joyce Jones.
Telephone: (202) 586-2090.

It is also on the WWW at:
http://www.ms.ornl.gov/cfcc/doe2076.htm

Infrared Burners

(Continued from page 5)

> "MTI has developed a fabrication process for the porous CFCC IR burners using both chopped and continuous fibers.

The key event during the pyrolysis cycle is the crystallization of the amorphous fiber to the garnet structure. This conversion generally occurs between 850° and 900°C. The garnet fibers generally exhibit a grain size of between 0.7 and 1.0 µm after firing.

Grain size

This grain size is generally considered too large to yield high-strength fibers. Fibers with a grain size less than 0.5 µm would be satisfactory, but a size of 0.2 µm is most desired to achieve high strengths. MTI and 3M have determined that dopants are most likely required to achieve these smaller grained microstructures. MTI has doped YAG fibers with chromia (up to 1.6 mole percent substitution of the alumina) and achieved some reduction in the resulting grain size.

Fig. 5. Vacuum winding of CFCC IR burner element.

MTI has developed a fabrication process for the porous CFCC IR burners using both chopped and continuous fibers. The burners are fabricated using the vacuum winding process that MTI developed for its hot-gas filters.

The process involves filament winding of continuous fibers (Nextel 610) while simultaneously vacuum forming a chopped fiber slurry onto the winding mandrel as shown in Fig. 5.

Preliminary IR emitters were fabricated using Saffil chopped fiber as a substitute for the ErHoAG fiber (to minimize raw material costs during the development stage and until the garnet fiber is available in larger quantities). As the rare-earth garnet fibers are developed, they will be incorporated into the vacuum winding process to produce a controlled spectrum IR emitter.

Figure 6 shows a MTI CFCC burner element being operated in an original equipment manufacturer-supplied burner segment.

The MTI CFCC emitters will be compared to a metal gas-fired IR burner at the Institute of Paper Science and Technology (IPST). A Krieger IR burner system is being installed on the IPST drying test facility to perform this comparison.

This facility utilizes an instrumented section of paper web that travels at up to 3000 ft/min to simulate paper production drying conditions. Thermocouples installed through the thickness of the paper web will be used to compare the temperature profiles and heating rates produced by the metal and CFCC burner systems.

Georgia Tech will characterize the emittance of the two burner systems using a spectral radiometer.Δ

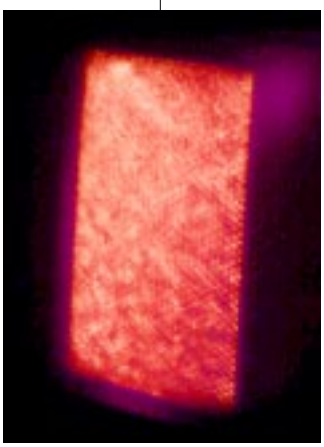

Fig. 6. Gas-fired CFCC IR burner.

10000-hour liner test

(Continued from page 3)

mal and air-coupled ultrasonic images were able to detect damage progression in the outer liner after 0, 2258, and 5016 hours of exposure to engine conditions.

Based on the evaluation of the CFCC liners after the first two field tests, it was evident that the SiC/SiC liners must be protected with EBCs for increased durability. Since the middle of 1998, Solar and P&W have worked jointly to improve and optimize EBCs developed under the National Aeronautics and Space Administration's enabling propulsion materials program, and apply them on CSGT liners.

After extensive testing in the ORNL furnace, the most promising EBCs were selected for application on seal-coated SiC/SiC liners for the current field-engine test at Texaco, which began in April 1999 (Fig. 4). The inner liner in this test is a Hi-Nicalon/SiC MI component. The outer liner is a Hi-Nicalon/Enhanced-SiC CVI part. Both liners were manufactured by ACI. EBCs were applied to only the working surfaces of the two liners.

Manufacturers

The Hi-Nicalon/SiC MI inner liner used in the 8000-h Malden Mills test, which began in the middle of August 1999, was made by BFG. The Hi-Nicalon/Enhanced-SiC outer liner was manufactured by ACI.

EBCs were applied to only the working surfaces of the two seal-coated liners. The liners have already accumulated more than 3000 h of exposure to combustion environment. It is planned to continue the current field test for at least an additional 1000 h.

For more information on the CSGT program, please contact Jeffery Price at 619-544-5538 or send e-mail to Price_Jeff_R@solarturbines.com.

Acknowledgement

This work was performed under DOE Contract DE-AC02-92CE40960. We are grateful to Stephen Waslo (Technical manager, DOE Chicago Operations), and Patricia Hoffman (Program manager, DOE Office of Industrial Technologies) for their support and guidance.Δ

"Based on the evaluation of the CFCC liners after the first two field tests, it was evident that the SiC/SiC liners must be protected with EBCs for increased durability.

Fig. 4. SiC/SiC CFCC liners protected with environmental barrier coatings.

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

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***CFCC News* URL**

http://www.hsrd.ornl.gov/cfcc/newshome.html

A COMMUNICATION OF THE CONTINUOUS FIBER CERAMIC COMPOSITE PROGRAM

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

Calendar

1999 Materials Solutions Conference & Exposition.
November 1–4, 1999, Convention Center, Cincinnati.
URL: http://www.MaterialsSolutions.com/mat-sol/

Advanced Turbine Systems Annual Review.
November 8–10, 1999, Pittsburgh Hilton & Towers, Pittsburgh.
A forum for energy experts from industry and government to discuss technical progress, energy policy and regulations, and university-based research.
For registration information, contact: FETC Conference Services, U.S. DOE, Federal Energy Technology Center, P.O. Box 10940, Pittsburgh, PA 15236-0940.
Telephone: (412) 386-4933 or (412) 386-4763
E-mail: barbish@fetc.doe.gov

ACerS Refractory Ceramics Division.
November 14–16, 1999, Lawrence Convention Center, Pittsburgh.
Joint meeting with the Iron and Steel Society's Electronic Furnace Conference.
URL: http://www.acers.org/meet/schedule.html

ACerS: 24th Annual Cocoa Beach Conference & Exposition.
January 23–28, 2000, Doubletree/Hilton Hotels, Cocoa Beach, Florida.
An international conference on engineering ceramics and structures.
URL: http://www.acers.org/meet/schedule.html

ACerS: 102nd Annual Meeting & Exposition.
April 30–May 3, 2000, St. Louis, Missouri.