CFCC News

Sponsored by the U.S. Department of Energy Office of Industrial Technologies

CONTINUOUS FIBER CERAMIC COMPOSITE MATERIALS

May 1988 • No. 10

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New Logo

Our new *CFCC News* logo was provided by DOE to represent the CFCC Program. The logo is one of a unified series developed to represent "Industries of the Future."

Thermal Characterization

An NDE Method for CFCCs

By J. G. Sun and W. A. Ellingson,
Argonne National Laboratory

As part of the Nondestructive Characterization project, Argonne National Laboratory (ANL) has developed a thermal imaging system for characterization of CFCC materials and nondestructive evaluation (NDE) of CFCC components.

A data base was established for thermal properties of CFCC materials, and experience was gained on testing components of various sizes and shapes.

An important element in this development was collaboration with industrial partners Dow Corning, DuPont Lanxide Composites (DLC), General Electric (GE), Solar Turbines, Babcock and Wilcox (B&W), Textron, and 3M.

Thermal Imaging System

Figure 1 shows a schematic of the thermal imaging system. It can be operated in a back-flash mode (shown in Fig. 1) to measure through-thickness thermal diffusivity or in a front-flash mode to detect defect depth.

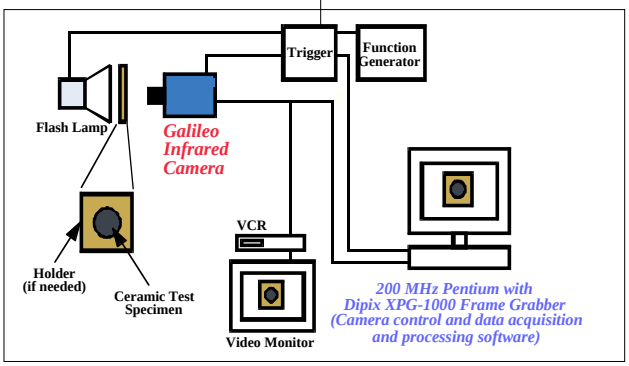

Fig. 1. Schematic of the thermal imaging system.

In the thermal diffusivity measurement, the thermal impulse from the flash lamp is applied on the back surface, and the temperature increase on the front surface is monitored by the infrared (IR) camera.

The rate of temperature rise is related to the through-thickness thermal diffusivity of the material. A defect (delamination, void, crack, etc.) is usually detected as a low diffusivity. In the front-flash test, both the flash lamp and the camera face the front surface of the sample. After the flash, the surface temperature variation is monitored by the camera.

Defect Detection

A delamination or low-porosity defect will show up as a low-diffusivity region on the surface because such defects restrict heat flow into the sample. The hot spot will show earlier if the defect depth is shallow and later if the depth is deep. Therefore, both thermal imaging methods can detect defects. The thermal diffusivity method may show the severity and depth of the

(*Continued on next page*)

defects. Improvement in the front-flash method is continuing.

The objectives of this research include providing data useful for process development and reliability, detecting and assessing damage and repairs, evaluating joining quality, and providing data for life prediction codes. Typical results are presented in the following. Most of the thermal diffusivity measurements were correlated and confirmed with other NDE methods such as X-ray tomography and ultrasonic imaging as well as with destructive testing.

> *"The objectives of this research include providing data useful for process development and reliability, detecting and assessing damage and repairs, evaluating joining quality, and providing data for life prediction codes."*

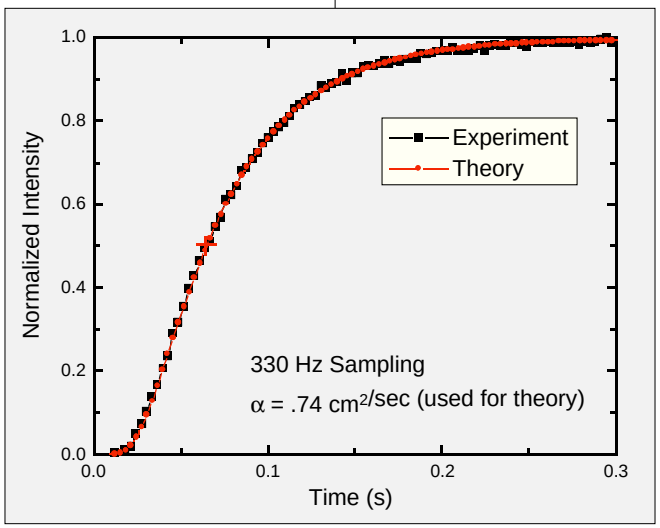

Fig. 2. Theoretical and experimental single-pixel history data.

Accuracy

The accuracy of the thermal diffusivity measurement was determined with a standard graphite specimen from the National Institute of Standards and Technology (NIST). Its thermal diffusivity value was reported to be 74 to 76 mm^2/s. Measured single-pixel history with the theoretical curve is shown in Fig. 2. The mean diffusivity value was found to be 72 mm^2/s, and the deviation was attributed to variations in specimen thickness. For CFCC materials examined to date, the thermal diffusivity ranges from 0.5 to 10 mm^2/s.

The IR camera is capable of obtaining ~50 µm/pixel resolution with a standard lens. Figure 3 shows a thermal diffusivity image of a B&W oxide-based CFCC cylinder with intentionally embedded defects. A defect size of 4 mm square can be easily identified.

Thermal diffusivity data have been measured for several CFCC materials: (1) SYLRAMIC™ composites fabricated by polymer impregnation process (PIP) from Dow Corning, (2) SiC/SiC composites produced by chemical vapor infiltration from DLC, (3) melt-infiltrated Silcomp™ composites from GE, and (4) NBSC from Textron.

Figure 4 shows diffusivity as a function of sample density for Dow Corning and DLC materials. The diffusivity value for the GE material is ~10 mm^2/s. Such data may provide a method to obtain density variation in a component.

Processing Information

A SYLRAMIC™ panel of 12 X 24 cm was characterized against PIP processing cycles. Figure 5 shows thermal diffusivity images of the 12 X 24 cm panel processed with 1, 5, 10, and 15 cycles, respectively. These series of images show the diffusivity increase (lighter gray scale) due to increased density in the panel and, in addition, show the progressive repairing of initially delaminated (dark) regions. The repairing of an impact-damaged SYLRAMIC™ panel was also character-

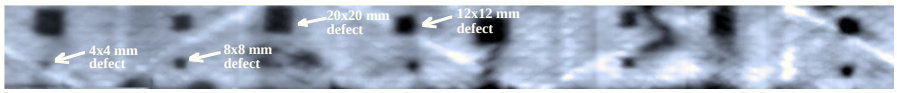

Fig. 3. Thermal diffusivity image of B&W cylinder showing multiple defects as dark regions.

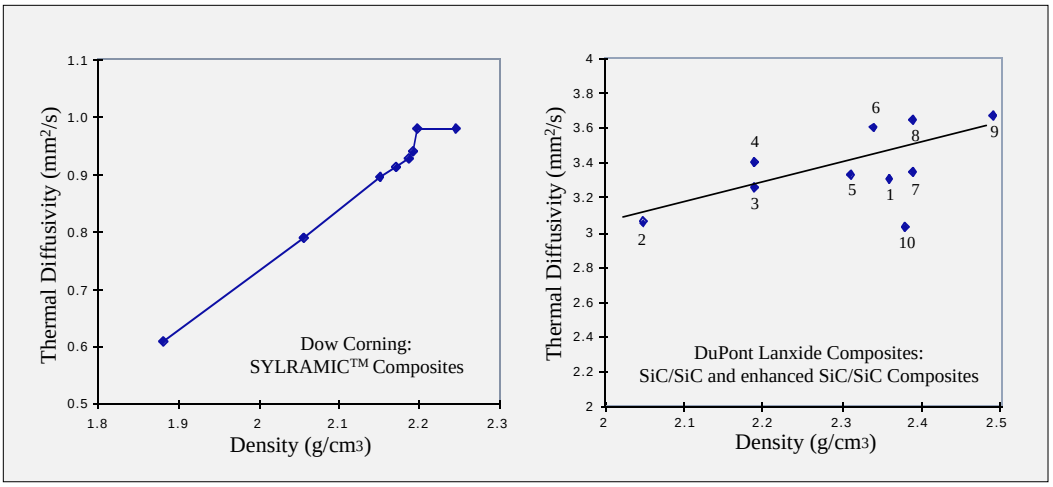

Fig. 4. Thermal diffusivity as a function of density for Dow Corning and DLC materials.

ized with through-thickness diffusivity and front-flash measurements as shown in Figs. 6 and 7, respectively.

Operation Induced Damage

Figures 8a and 8b show the thermal diffusivity images of a DLC SiC/SiC subscale combustor liner before and after a test of ≈ 25 h in a high-temperature test rig, respectively. The image in Fig. 8a has horizontal and vertical lines marked on the surface.

diffusivity region. In Fig. 9, the thermal diffusivity image of a Dow Corning subscale liner after a 100-h test run showed two cracks that were later identified and confirmed by Dow Corning with destructive methods.

Thermal shock damage

Thermal shock damage has also been studied using SiC/SiC coupons (3.8-cm squares) provided by the

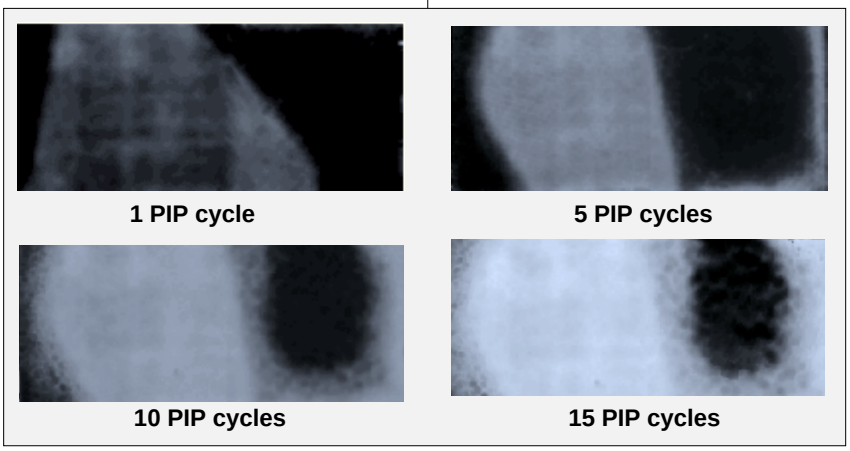

Fig. 5. Thermal diffusivity images of a SYLRAMIC™ panel.

The image in Fig. 8b shows some surface patterns due to the flaking of the surface coating. Both images show a large region with low thermal diffusivity, presumably a delaminated region. By comparing the two images, it is apparent that after the high-temperature test, there is a significant increase in the area of the low-

University of Cincinnati. These were quenched in water with quenching temperature difference ΔT up to 1000°C. Figure 10 shows diffusivity images of a coupon before and after quenching with ΔT = 1000°C. The thermal shock damage is severe at the coupon edges. The correlation of

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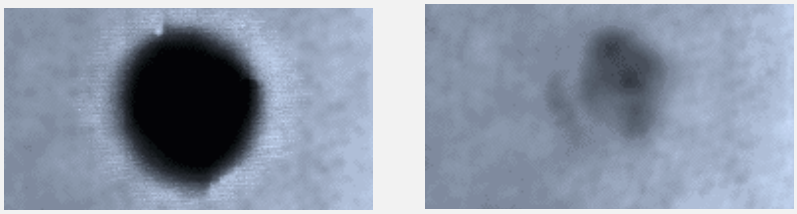
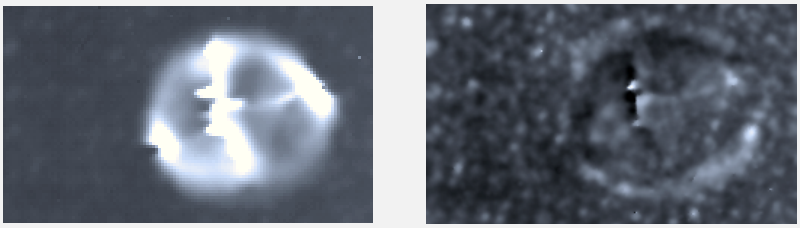
> *"These series of images show the diffusivity increase (lighter gray scale) due to increased density in the panel and, in addition, show the progressive repairing of initially delaminated (dark) regions."*



Before repair After repair

Fig. 6. Thermal diffusivity images of impact-damaged panel A2.



Before repair After repair

Fig. 7. Front-flash thermal images (at 0.33 s) on non-impact surface of panel A2.

> *"Low-diffusivity regions may suggest incomplete bonding at the joints."*

diffusivity profiles along a diagonal line is shown in Fig. 11. These data suggest that thermal shock damage is more severe at edges.

Joining

Thermal imaging methods may also be used to characterize the quality of joints. Figure 12 shows two sets of thermal diffusivity images of CFCC/CFCC specimens. One set was produced by reaction-bonded SiC at Idaho National Engineering Laboratory and the second set with PIP processing at Dow Corning. Low-diffusivity regions may suggest incomplete bonding at the joints.

Conclusion

As part of the NDE project a rapid infrared thermal imaging system developed at ANL provides accurate measurements of thermal diffusivity values for CFCC samples. The system is effective for measuring manufacturing- and operation-induced defects in CFCC components. The method can, therefore, be used for reliable characterization of CFCC materials and provide data for life prediction of CFCC components.▲

(a)

(b)

Fig. 8. Thermal diffusivity images of a DLC SiC/SiC liner, (a) pretest and (b) after ≈ 25-h test.

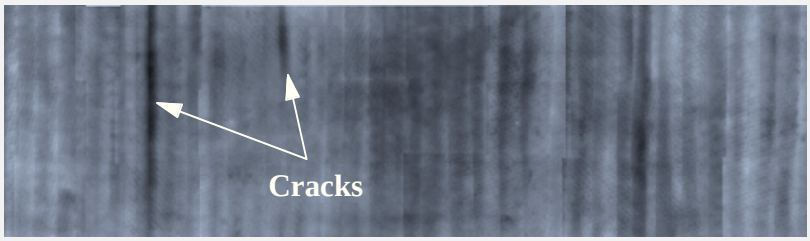
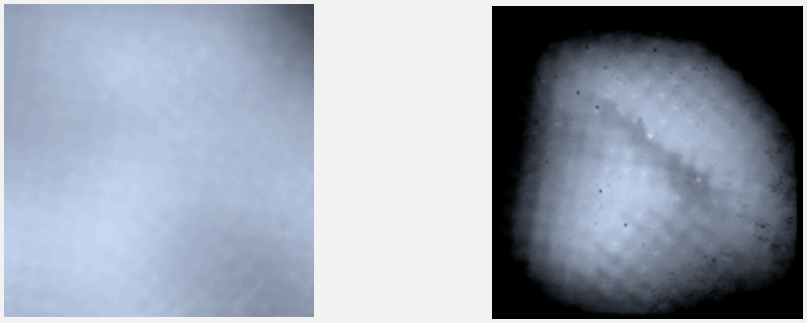


Fig. 9. Thermal diffusivity images of a Dow Corning subscale liner after 100-h test.



Quenched | Unquenched

Fig. 10. Thermal diffusivity images of a SiC/SiC coupon under thermal shock test.

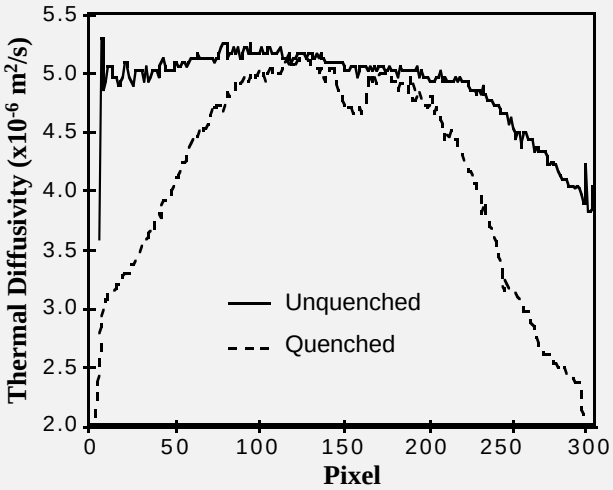
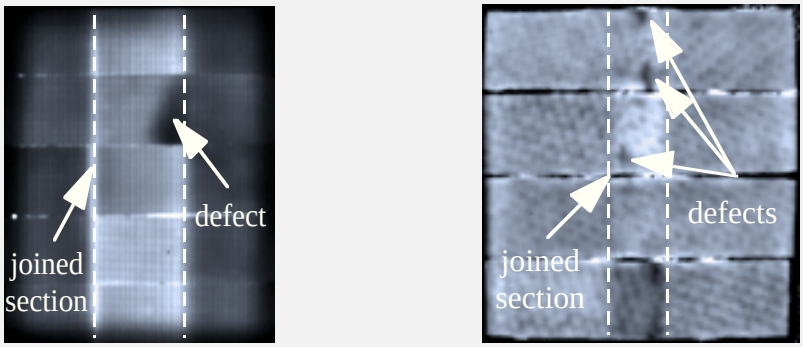


Fig. 11. Thermal diffusivity profiles along a diagonal line of images in Fig. 10.



Reaction bonded joints | PIP processed joints

Fig. 12. Thermal diffusivity images of joint specimens.

> *"The system is effective for measuring manufacturing- and operation-induced defects in CFCC components. It can, therefore, be used for reliable characterization of CFCC materials and provide data for life prediction of CFCC components."*

Into the Future

CFCCs Will Benefit Industries of the Future

By Jim Wessel, Wessel & Associates

Commercialization. Is there a sweeter sounding word to the ears of a material supplier? Probably not. Commercialization is a goal whose realization has eluded CFCC suppliers for many years. But thanks to a new Department of Energy (DOE)-sponsored activity, that goal now seems attainable.

The DOE "Conference Marketing Plan for the CFCC Program" provides specific guidance for marketing the features and benefits of CFCC materials to major industries. One objective of this plan is to create a demand for CFCC technology by end users.

Currently, many end users are unaware of the increased benefits and enhanced value of equipment constructed with CFCC materials. Once they become aware, these users are likely to ask their equipment suppliers about CFCC materials and thereby create an expanding market. Thus, the first step toward achieving this objective is to make end users and equipment manufacturers aware of CFCC materials.

> "Currently, many end users are unaware of the increased benefits and enhanced value of equipment constructed with CFCC materials."

New Uses

Another objective of this program is to identify additional uses for CFCC materials. Marketing the features and benefits of CFCC materials to additional potential users will result in other uses.

The Conference Marketing Plan can accelerate the achievement of both objectives. The plan recommends creating exhibits and literature for display and handout at major industry conferences. The first task, however, is to identify the best conferences.

Synergy

The conferences of the "Industries of the Future" were chosen to create synergy with DOE's CFCC program. In that program, DOE's Office of Industrial Technologies encourages energy- and resource-intensive industries to adopt advanced technologies.

Participants include the aluminum, chemical, glass, forest products, metalcasting, and steel industries. Materials technology crosscuts all of these industries.

The National Association of Corrosion Engineers (NACE) annual meeting was

Fig. 1. Debbie Haught at the CFCC booth prior to the NACE meeting opening.

chosen as the initial conference because these engineers struggle with harsh environments that eliminate other materials. Attendees are mostly from the chemical and refining industries. Last year more than 5000 people attended this meeting.

Keeping their audience in mind, Bill Long, Mike Karnitz, Jim Wessel, Dave Cotrell and Penny Humphreys—with advice from Debbie Haught (CFCC program manager), CFCC principal investigators, and the Steering committee—designed an exhibit and supporting literature that would be of the greatest benefit to chemical engineers and equipment suppliers.

The result was a display that featured four chemical industry applications for CFCC materials, plus literature describing two other potential uses and general CFCC literature that enabled engineers to conceive of new uses. The display also included CFCC photos and components supplied by CFCC contractors.

San Diego Conference

The conference was held March 23–25, 1998 in San Diego's convention center. Booth workers included Andy Szweda, Tom Foltz, Richard Goettler, Debbie Haught, Bill Long, and Jim Wessel. As new exhibitors, we were fortunate to be located adjacent to popular large exhibits.

This was the debut of CFCC to a broad industry audience. Our typical visitor paused at our booth with a puzzled look on their face. "Have you heard of CFCC materials?" our booth attendant asked. "No" was the usual response. What a great response for the ensuing explanation by our booth worker. Other visitors stopped because they recognized the corporate logos of the CFCC contractors. The CFCC brochure and company industrial application sheets were popular pieces of literature.

Many potential customers stopped by to explain problems. Some described problems potentially solvable by using CFCC materials. They were given advice, appropriate literature, application and contact information (collected on a specially designed form) and, where needed, a promise to send additional information or have the appropriate person contact them. All CFCC principal investigators will have an opportunity to make those contacts.

Valuable Lessons

We learned a number of valuable lessons at this conference. The quality of visitor discussions would improve if a CFCC-related paper were presented at the adjoining technical session. This paper should familiarize the audience with basic CFCC information, resulting

(Continued on page 11)

> *"We learned a number of valuable lessons at this conference. The quality of visitor discussions would be improved if a CFCC related paper were presented at the adjoining technical session."*

New on the CFCC Web Site

The following information is available through the CFCC Web Site **(http://www.ms.ornl.gov/cfcc/)**

- CFCC Brochure that describes the CFCC program. **(http://www.ms.ornl.gov/cfcc/brochure/cfccbroch.pdf)**
- Industrial Application Sheets showing the benefits of using continuous fiber ceramic composites in the applications below. **(http://www.ms.ornl.gov/cfcc/brochure/ind_appl.htm)**
  1. Pipe Hangers for Oil Refineries,
  2. Reformer Efficiency,
  3. Industrial Gas Turbines,
  4. Improved Filter Life,
  5. Heat Exchanger Tubes, and
  6. Improved Radiant Burner Efficiency.

Working Group

Improvements in Materials, Cost Effectiveness, Innovation Lead to 1997's CFCC Accomplishments

By Merrill Smith and Debbie Haught, CFCC Progam Managers

Progress in the CFCC program by both industry and research groups during the 1997 fiscal year was outlined during the Working Group Meeting in October by DOE CFCC program managers Merrill Smith and Debbie Haught. The year's accomplishments included improvements in both materials and processes. Their report follows.

Materials & Process Improvements

• Demonstrated 90% improvements in micro crack yield point at RT of Sylramic S200 with use of Sylramic SiC fiber (Dow Corning)

• Improved near net shape fabrication processes for valve guide blanks and reduced ID/OD machining stock by >20% (AlliedSignal)

• Developed resin-derived interface carbon coating, with cost one-tenth of that for the CVI process (DuPont Lanxide)

• Completed 6600-h stress rupture test that demonstrated reproducible low-cycle fatigue behavior of DIMOX material (Du Pont Lanxide)

• Developed CFCC cost model for polymer impregnation process (PIP) that suggests 25% reduction in manufacturing costs are possible (Dow Corning)

• Developed promising scheelite ($CaWO_4$) fiber coating for oxide-oxide composites (McDermott)

• Optimized fiber architecture for filament-wound CFCC components. Ordered new 5-axis filament winder (Textron)

• Initiated efforts in low-cost fiber-reinforced thin sheets to produce CFCCs at $100/lb (Du Pont Lanxide)

Environmental Durability

• 4000-h exposure testing in direct-coal-burning boiler at TVA Gallatin Station was completed for Nicalon/E-SiC tubes. The samples showed no evidence of corrosion or strength loss after exposure (1628°F avg) (DuPont Lanxide)

• Sylramic S200 coupons exposed in crude unit furnace for 8424 h still retained greater than 60% of original strength (Dow Corning)

Application-Specific Progress

• Hot gas filter testing at Foster Wheeler pilot-scale 10-MW pressurized fluidized-bed combustion facility in Karhula, Finland, has begun (67 h testing to date) (McDermott)

• Proof of concept for CFCC IR radiant burner (based on TPV emitter technology) was demonstrated (McDermott)

• Filter materials from 3M, Amercom, DuPont Lanxide, McDermott, Smart Ceramics, Techniweave, Pall Corporation, and Dow Corning have been tested for 24 h in a series of fluidized-bed reactor simulators in the furnace. Rupture tests of ring samples are under way

• Conducted simulated engine testing which demonstrated that CFCC valve guides caused less wear than both silicon nitride and cast iron guides (AlliedSignal)

> *"The year's accomplishments included improvements in both materials and processes."*

• Tested CFCC valve guides in Caterpillar gas-fired, diesel cycle engine for 224 h (AlliedSignal)

• Conducted 32,000 thermal cycle tests (2100°F face temperature) on SiC/SiC radiant burner screens with no failure (DuPont Lanxide)

• Solar Turbines field test (1850°F TRIT) completed 1000 h on CVI SiC combustor liners (DuPont Lanxide)

• CVI combustor liner survived 100-h test at Solar Turbines despite fabrication damage (delamination) (DuPont Lanxide)

• New shroud design was developed with reduced stress and better manufacturability (GE)

• Initiated coordinated effort between Virginia Tech, Solar Turbines and Oak Ridge National Laboratory (ORNL) to predict the service life of combustor liners for gas turbine engines based on MRLife code.

Factors that Limit Use of CFCCs

• Used electron microscopy along with mechanical property characterization to characterize effect of oxygen in BN interfaces for SiC/SiC composite systems. (Demonstrated oxygen contamination in BN degrades the mechanical reliability of these composites.)

• A micromechanical model was developed to predict the oxidation-induced stress-rupture behavior of CFCCs.

• CFCC environmental effects test facilities at ORNL were established and initial experiments in inert gases, air, and air and water vapor have been conducted at elevated temperatures.

• Instrumental in formalizing 7 full-consensus standard test methods that have been adopted by the American Society for Testing and Materials subcommittee C28.07 on Ceramic Matrix Composites

• Developed test to characterize the hoop strength of CFCC tubular components.

• The U.S. Advanced Ceramics Association conducted a comprehensive study of how advanced ceramics can be targeted in the seven Industries of the Future to improve energy efficiency and solve materials-related problems.▲

“Initiated coordinated effort between Virginia Tech, Solar Turbines and Oak Ridge National Laboratory (ORNL) to predict the service life of combustor liners for gas turbine engines based on MRLife code.”

Summary of Task 2 Review in Next Issue

The next issue of *CFCC News* will include a summary of the CFCC Supporting Technologies Program (Task 2) review, which was conducted March 18–19 at Oak Ridge, Tennessee.

The review specifically examined the interaction between the Supporting Technologies Program and the Industrial Contracts.

The last program review was discussed in the August 1997 issue of *CFCC News* (No. 9).▲

1997 Working Group Proceedings

The “Proceedings” (DOE/ORO 2060) of the October 1997 CFCC Working Group Meeting in Lake Tahoe, Oct. 6–7, 1997, is available on request.

DOE and DOE contractors can get a copy from The Office of Scientific and Technical Information, PO Box 62, Oak Ridge, TN 37831. For price information, call 423/576-8401.

The public can get a copy from the National Technical Information Service, U.S. Department of Commerce, 5285 Port Royal Rd., Springfield, VA 22161.▲

Hispanic Engineer Wins 1987 Achievement Award

Edgar Lara-Curzio, a development staff member in the Metals & Ceramics Division at Oak Ridge National Laboratory (ORNL), has been recognized for his "Outstanding Technical Achievements" by the 1997 Hispanic Engineers National Achievement Awards Conference in Houston.

Edgar Lara-Curzio

Speaking at the awards ceremony, U.S. Department of Energy Secretary Federico Pena acknowledged the achievements of three Hispanic scientists from the National Laboratories.

Lara-Curzio, a native of Mexico City, is part of the High Temperature Materials Laboratory research staff where he serves as principal investigator for DOE's Composites (CFCC) Program.

Lara-Curzio is widely recognized for his leadership in developing test standards for ceramic matrix composites and for investigating the effects of stress, temperature, and environment on the mechanical behavior of advanced ceramics and composites.

He is also a leader in developing models to predict the mechanical behavior, reliability, and performance of these materials.

Novel Facilities

The ORNL scientist has done important work in the design and construction of novel facilities for the thermomechanical evaluation of advanced materials.

In addition to his research and development efforts, Lara-Curzio is highly regarded for mentoring students and for his role in supporting U.S. industry and academia in the solution of materials problems through the High Temperature Materials User Program.

Awards

Hua-Tay Lin

Hua-Tay Lin has won a Science and Technology Agency Fellowship from Japan Science and Technology Corp.

A research staff member of the Structural Ceramics Group in the Metals and Ceramics Division of Oak Ridge National Laboratory, Lin was cited for his leadership and research in the high-temperature mechanical behavior of ceramics and ceramic composites.

Science Fellowship For ORNL Scientist

Lin is a member of the American Society for Metals International; the Minerals, Metals and Materials Society; and the American Ceramics Society.

He received Martin Marietta Energy Systems Technical Support Awards in 1993 and 1996 and an MMES Technical Achievement Award in 1991.

3rd Award for CFCC News

CFCC News has won its third award from the Society for Technical Communication. The Merit Award was given in the newsletter category of the group's 1997/1998 competition.

The newsletter is prepared by Gloria Caton, Managing Editor, and Tim Elledge, Designer/Editor/Writer, for the DOE Continuous Fiber Ceramic Composite Program/DOE Office of Industrial Technologies.

Research Notes

■ Advanced Ceramics in the Vision Industries

Researchers have completed a 12-month study to identify ways that advanced ceramics might be used in industries related to vision products and services. The work was sponsored by DOE–U.S. Advanced Ceramics Association (USACA).

The ceramics considered in the study include monolithics and composites. Refractories and coatings were also included but to a lesser extent.

Vision industries include work in steel, metal casting, aluminum, refineries, chemicals, forest products, and glass. Data were collected from DOE, ORNL, and other sources. They were used to assess current and past DOE programs in advanced ceramics that have resulted in technology contributions leading to increased energy efficiency and pollution prevention in these industries.

Each of the industries was reviewed to learn where advanced ceramics are being used and where the greatest benefits could be achieved from their increased usage. The study also examined barriers to using ceramics in the marketplace. Potential applications and people to contact in the industries were catalogued.

A preliminary overview of the results was presented last fall at a meeting that included speakers from each of the industries. The final report is being completed. An article reviewing the results is planned for the next issue of the CFCC newsletter. (From Doug Freitag and David Richerson)

Into the Future with CFCC Materials

(Continued from page 7)

in greater progress at a subsequent booth visit.

We will have more hardware at future meetings. CFCC components are good conversation pieces that help potential users get application ideas. Also, our visitors' low level of ceramic knowledge indicates that we need to provide more basic information on ceramic composites.

Exhibiting at the NACE conference was a good idea. We educated a number of engineers about the uses and benefits of CFCC materials.

The challenge is both to select the next conference as well as to develop a way to reinforce the missionary work started with the chemical engineers. The latter goal will be pursued by a workshop adjacent to the June 1998 NACE meeting in Houston.

Other Conferences

Other Industries of the Future conferences are appropriate for CFCC information dissemination. Another important meeting is the 1999 Annual National Design Engineering Show and Conference where 60,000 attendees look for new materials and new hardware designs. These will all be evaluated and selected for maximum benefit.

The NACE meeting indicated the limited awareness of ceramic composites and the need to educate potential customers. The Conference Marketing Plan for the CFCC Program will help jump this hurdle and speed CFCC suppliers on their way to commercializing these valuable materials.▲

“The challenge is both to select the next conference as well as to develop a way to reinforce the missionary work started with the chemical engineers.”

CFCC News

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

National Association of Corrosion Engineers (NACE) Workshop

June 29–30, 1998, Nassau Bay Hilton, Houston, Texas.
Ceramics: Cutting Edge Solutions for Corrosion Control.
This meeting is being organized by NACE with help from the DOE Office of Industrial Technologies and the support of U.S. Advanced Ceramics Association. Contact: Shelley Leavitt Nadel, NACE Director of Public Affairs and Technical Activities.
Telephone (281) 228-6245, Fax (281) 228-6345
E-mail: shelley@mail.nace.org

CFCC Working Group Meeting

July 1, 1998, Nassau Bay Hilton, Houston, Texas.
The Working Group meeting follows the NACE Workshop.
Contact: Shirley North, Metals and Ceramics, ORNL.
Telephone (423) 574-8860, Fax (423) 241-0411
E-mail: northsa@ornl.gov

Fifth International Conference on Composites Engineering

July 5–11, 1998, Las Vegas.
Meeting information is available at
http://www.uno.edu/~engr/composites.html