CERAMIC RELIABILITY FOR MICROTURBINE HOT-SECTION COMPONENTS

Reliability Evaluation of Microturbine Components

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Objective

Evaluate and document the long-term mechanical properties of very small specimens machined from ceramic components (e.g., blades, nozzles, vanes, and rotors) in as processed and after engine testing at ambient and elevated temperatures under various controlled environments. This work will allow microturbine companies to verify mechanical properties of components and apply the generated database in probabilistic design and lifetime prediction methodologies. The work also provides a critical insight into how the microturbine environments influence the microstructure and chemistry, and thus the mechanical performance of materials.

Highlights

Evaluation of mechanical properties of a Kyocera SN281 silicon nitride microturbine rotor was completed during this reporting period. The SN281 microturbine rotor was manufactured by Kyocera Industrial Ceramics Corporation, Vancouver, Washington and was acquired from UTRC. Biaxial tests were carried out at room temperature to generate the mechanical properties for airfoils, typically subject to high temperatures and stresses during operation, with as-processed surfaces. Data were also generated for biaxial disks as well as ASTM MOR test bars machined from the hub region for baseline. The database has been provided to UTRC and Kyocera for probabilistic component life prediction and property verification, respectively.

Technical Progress

Studies of dynamic fatigue for NT154 silicon nitride (Saint-Gobain Ceramics & Plastics, Northboro, MA) were continued during this reporting period. Saint-Gobain was awarded a contract by DOE under Microturbine Materials Program to re-develop NT154 silicon nitride for hot-section components for advanced microturbines. The purpose of this study is to generate a database for verification of mechanical performance and also for probabilistic component design and life prediction. The NT154 silicon nitride billets were fabricated under the Phase I contract, and MOR bars were longitudinally machined per the revised ASTM C116 standard with 600 grit surface finish. Half of the billets were machined and tested with as-processed surface. The bend bars with as-machined surface are designated as NT154-AM, and those with as-processed surface are designated as NT154-AP in the present study. The dynamic fatigue tests were carried out at 20, 850, and 1204°C and at stressing rate of 30 and 0.003 MPa/s in air per ASTM C1465. The 30

MPa/s is used to evaluate the inert characteristic strength as a function of temperature, and 0.003 MPa/s is applied to measure the slow crack growth (SCG) susceptibility at temperature.

Test results at 0.003 MPa/s showed that the NT154-AM material exhibited 21-38% decrease in inert characteristic strength as compared those generated at 30 MPa/s under the same test temperatures (Table 1, and Fig. 1, 3, and 5). The NT154-AP material exhibited little change in inert strength when tested at 0.003 MPa/s (Table 1, and Fig. 2, 4 and 6). The test results of NT154-AM indicated that the bulk material exhibited low fatigue exponents under the temperature range employed, indicting high susceptibility to slow crack growth (Fig. 7). On the other hand, the NT154-AP material exhibited very high fatigue exponents, which are in sharp contrast to the bulk material (Fig. 8). The detailed SEM examinations will be carried out on fracture surfaces of selected bend bars to understand the cause of low fatigue exponents of bulk materials.

Table 1. Summary of uncensored Weibull and strength distributions for Saint-Gobain NT154 silicon nitride specimens with as-processed, designated as NT154-AM, and as-machined, designated as NT154-AM (longitudinally machined per the revised ASTM C1161 standard).

Material	# of Spmns. Tested	Stressing Rate (MPa/s)	Temp. (°C)	Uncens. Weibull Modulus	± 95% Uncens. Weibull Modulus	Uncens. Chrctstic Strength (MPa)	± 95% Uncens. Chrctstic Strength (MPa)
NT154-AM	12	30	20	21.42	13.11, 31.73	929	901, 957
NT154-AM	12	0.003	20	9.62	5.95, 14.07	737	688, 787
NT154-AP	15	30	20	4.72	3.02 6.85	574	506, 645
NT154-AP	15	0.003	20	6.81	4.42 9.68	532	488, 577
NT154-AM	12	30	850	10.16	6.12, 15.41	777	727, 827
NT154-AM	12	0.003	850	28.39	17.05 43.33	527	515, 539
NT154-AP	15	30	850	6.0	3.75, 8.86	493	447, 541
NT154-AP	15	0.003	850	6.68	4.30, 9.60	470	430, 511
NT154-AM	12	30	1204	18.91	11.23, 28.98	708	683, 732
NT154-AM	12	0.003	1204	15.98	9.90, 23.55	439	421, 457
NT154-AP	15	30	1204	5.66	3.58, 8.38	514	463, 567
NT154-AP	15	0.003	1204	7.04	4.42, 10.41	458	421, 495

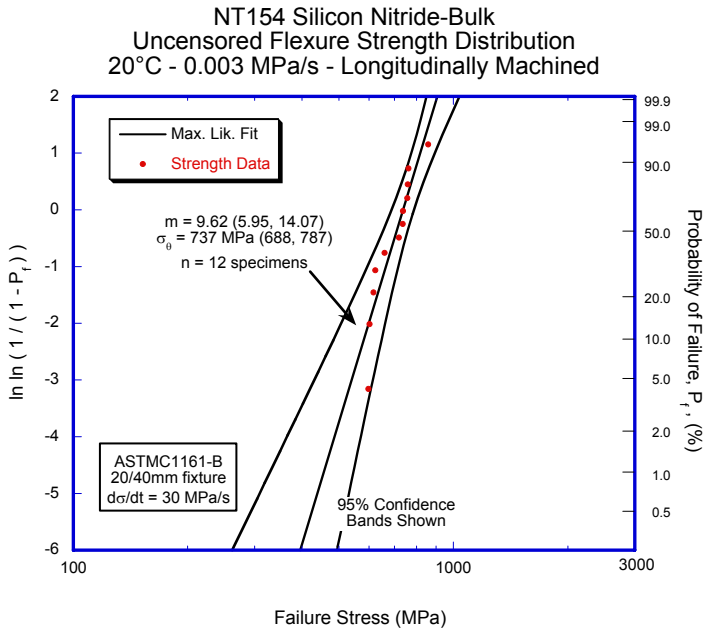

Figure 1. Uncensored flexure strength distribution at 20°C and 0.003 MPa/s of NT154 silicon nitride with as-machined surface (bulk material).

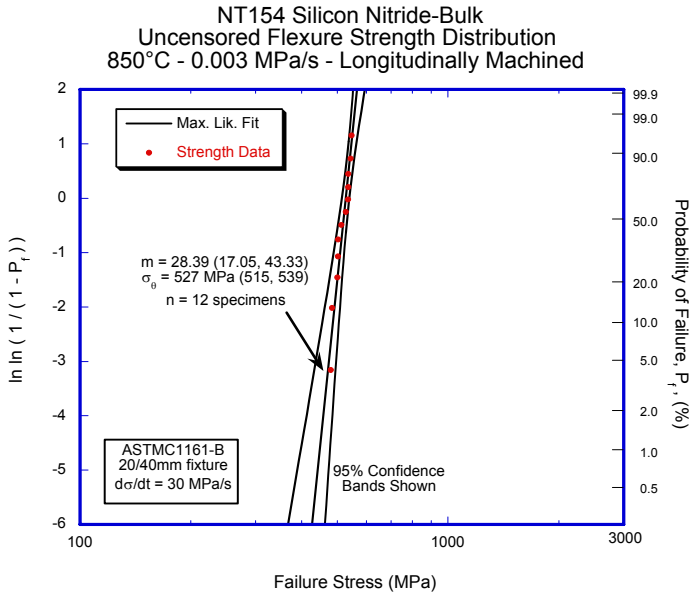

Figure 3. Uncensored flexure strength distribution at 850°C and 0.003 MPa/s of NT154 silicon nitride with as-machined surface (bulk material).

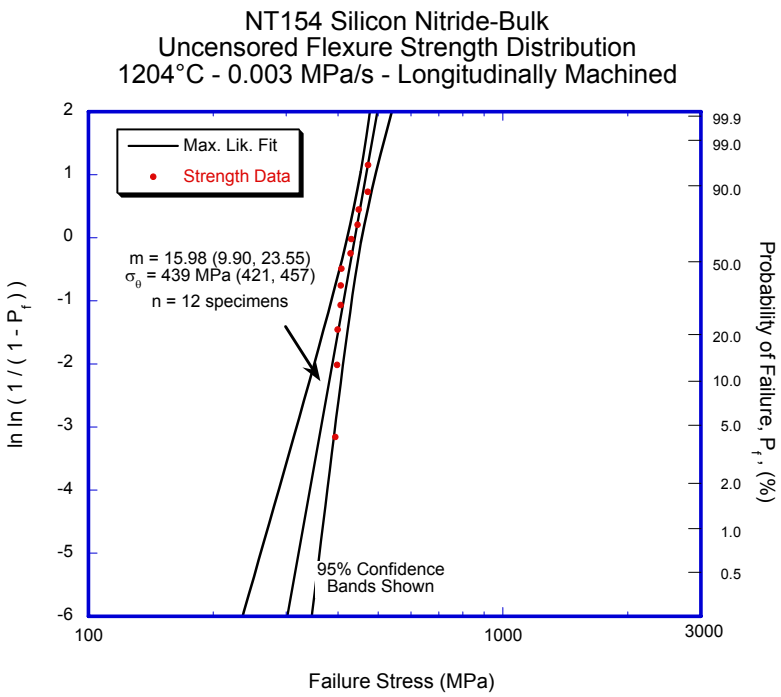

Figure 5. Uncensored flexure strength distribution at 1204°C and 0.003 MPa/s of NT154 silicon nitride with as-machined surface (bulk material).

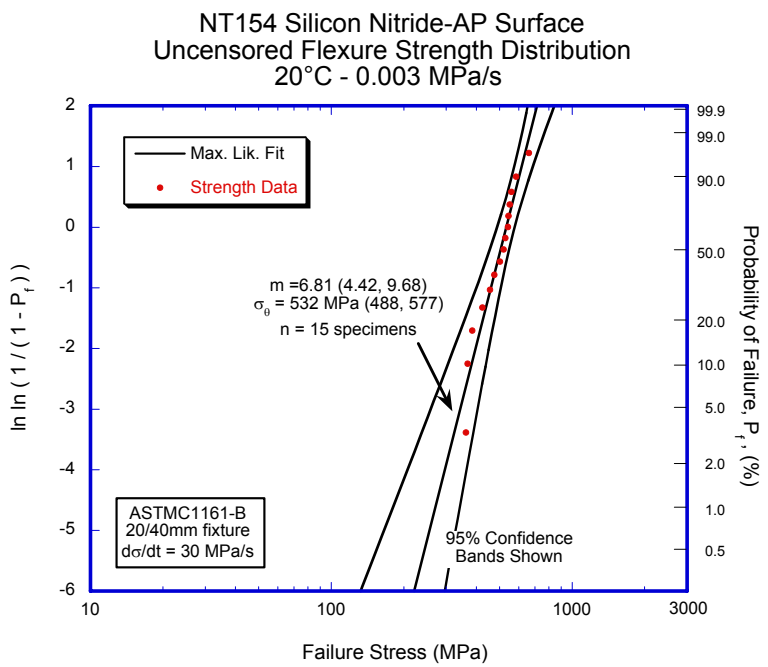

Figure 2. Uncensored flexure strength distribution at 20°C and 0.003 MPa/s of NT154 silicon nitride with as-processed surface.

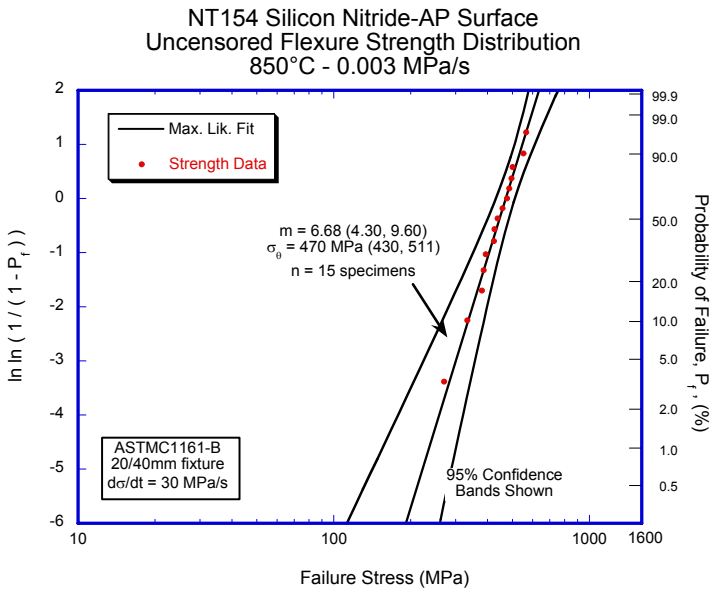

Figure 4. Uncensored flexure strength distribution at 850°C and 0.03 MPa/s of NT154 silicon nitride with as-processed surface.

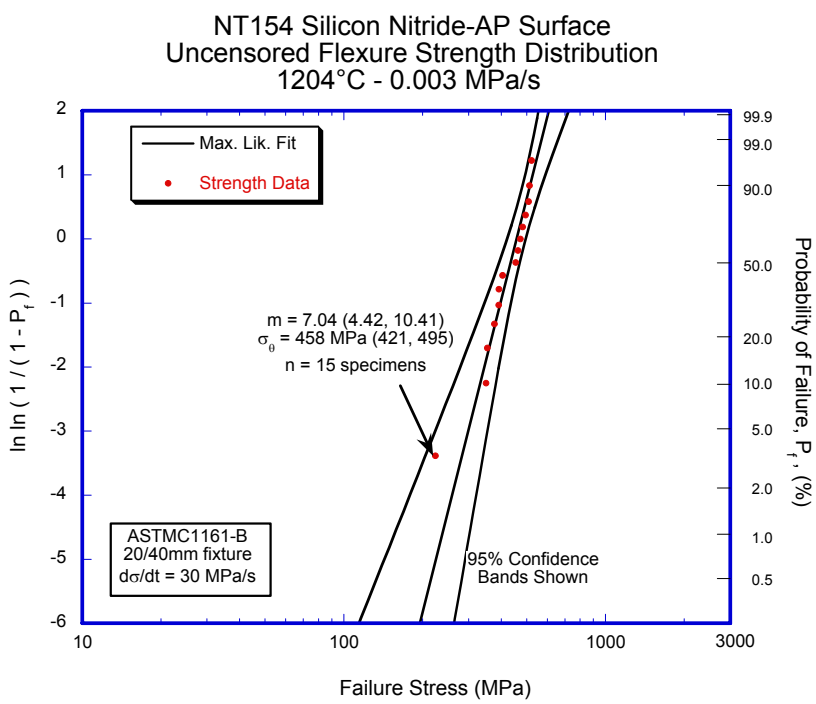

Figure 6. Uncensored flexure strength distribution at 1204°C and 0.003 MPa/s of NT154 silicon nitride with as-processed surface.

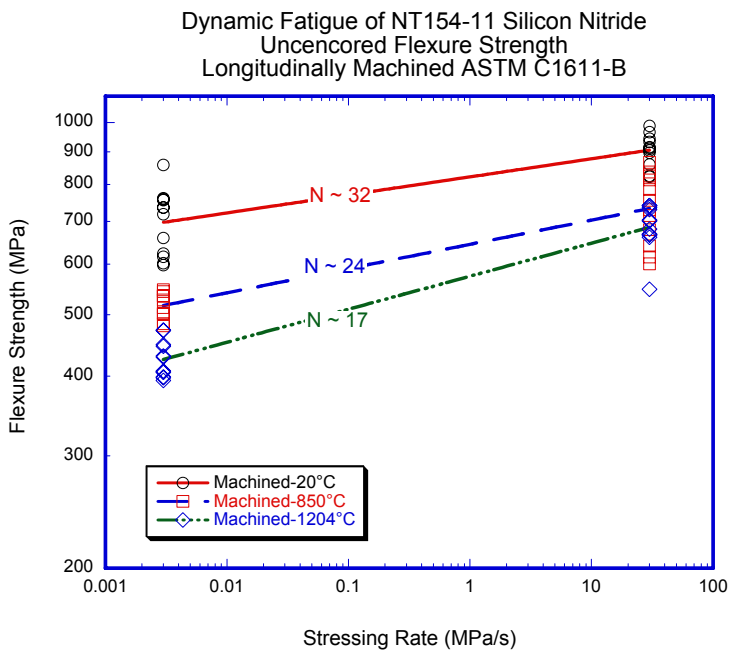

Figure 7. Flexure strength versus stressing rate for NT154 silicon nitride with as-machined surface (bulk material) at 20, 850, and 1204°C.

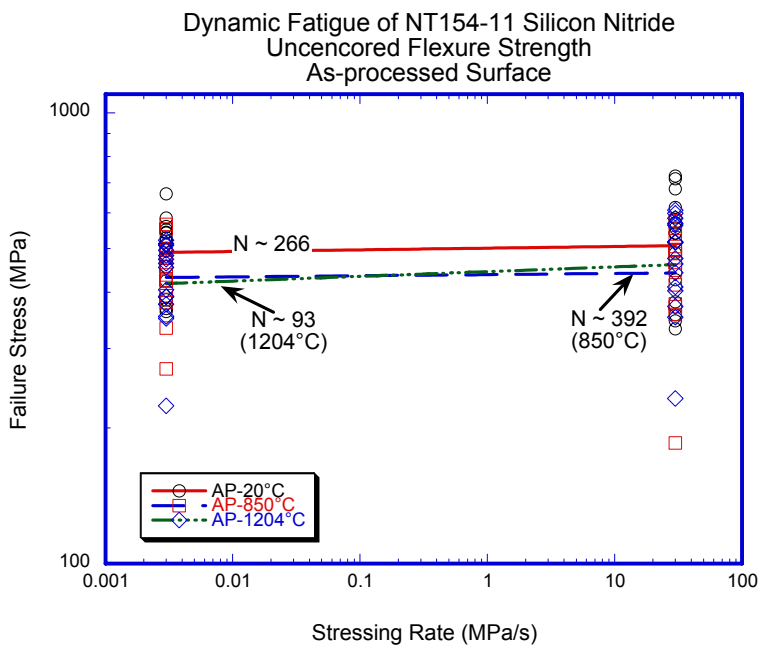

Figure 8. Flexure strength versus stressing rate for NT154 silicon nitride with as-processed surface at 20, 850, and 1204°C.

Evaluation of mechanical properties biaxial disks machined from the hub of Kyocera SN237 microturbine rotor, acquired from Ingersoll-Rand (IR), was completed during this reporting period. The data generated for samples from the hub will be used as reference. Figure 9 shows the uncensored strength distribution of biaxial disks extracted from the hub IR SN237 microturbine rotor. Mechanical results showed that SN237 biaxial specimens with machined surface exhibited a relative high characteristic strength of 1037 MPa with respect to the value (814 MPa) obtained for disks extracted from as-processed airfoils. Note that the samples from as-processed airfoils exhibited a higher Weibull modulus (m = 19.1) than that (m = 9.47) obtained from the hub region.

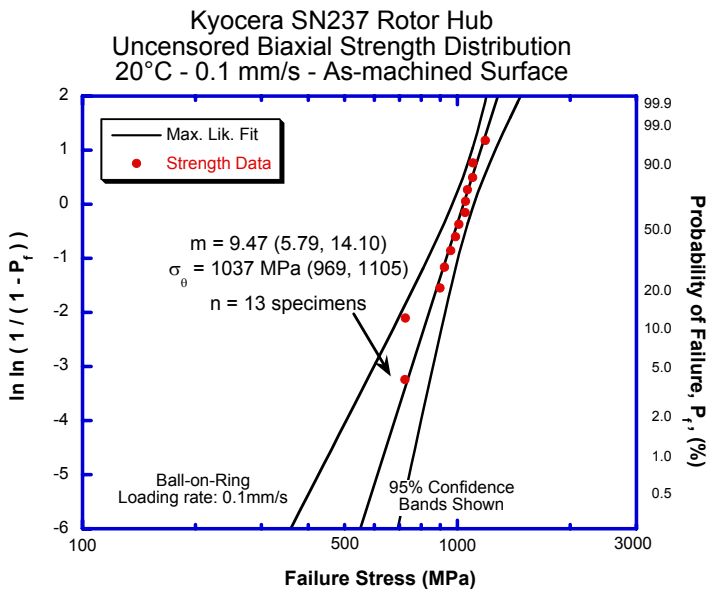

Figure 9. Uncensored flexure strength distribution at 20°C for biaxial disks machined from the hub of SN237 microturbine rotor.

Status of Milestones

1. Complete characterization of microstructure and mechanical properties for Ingersoll-Rand SN273 silicon nitride microturbine rotors by September 2003. Completed.

Industry Interactions

Monthly teleconferences with John Holowczak, Tania Bhatia, Venkata Vedula, and Gary Linsey at UTRC to discuss the mechanical database of Kyocera SN282 and Saint-Gobain NT154 silicon nitride for microturbine applications.

Communication with Vimal Pujari and Ara Vartabedian at Saint-Gobain Ceramics & Plastics on the updates of the testing status and results for NT154 silicon nitride materials manufactured during Taks I and II under Phase I contract.

Communication with Jim Kesseli at Ingersoll-Rand Energy Systems about the biaxial test results from the hub region for Kyocera SN237 silicon nitride microturbine rotor.

Communication with Russ Yeckley at Kennemetal on the microstructure and composition of SiAlON materials received.

Problems Encountered

None

Publications/Presentations

1. H. T. Lin, M. K. Ferber, J. R. Price, J. B. Kimmel, and M. van Roode, "Characterizations of Silicon Nitride Ceramic Components after Exposure to an Industrial Gas Turbine,"**Invited paper,** presented at the 5th International Meeting of Pacific Rim Ceramic Societies (PacRim5), September 29 – October 2, 2003, Nagoya, Japan.

Long-Term Testing in Water Vapor Environments

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Objective

The primary objective of this project is to evaluate the long-term mechanical and chemical stability of advanced materials of interest to the DER program. Currently the project is evaluating (1) structural ceramic, which are being considered for use as hot-section components in microturbines, and (2) thick thermal barrier coatings (TTBCs) being developed for thermal management in combustor liners used in industrial gas turbines. The structural ceramics effort focuses on the development and utilization of test facilities for evaluating the influence of high-pressure and high-temperature water vapor upon the long-term mechanical behavior of monolithic ceramics having environmental barrier coatings. In the case of the TTBCs, the primary focus is on the evaluation of changes in microstructure and thermal properties arising from long-term aging tests.

Highlights

Studies to evaluate the temperature sensitivity of recession of silicon carbide ceramics exposed in the steam injection rig were initiated.

Technical Progress

The effort to characterize the free-standing TTBCs was continued. These specimens, which were in the form of disks, nominally 10 mm in diameter and 1.17 mm thick, were aged for 100, 500, 1000, 2000 and 5000 h at temperatures of 1800, 1900, 2000, 2100, 2150, 2200, 2250, and 2300°F (982, 1038, 1093, 1149, 1177, 1204, 1232, and 1260°C). The following properties were measured for the samples aged for 2000 and 5000 h: hardness, thermal diffusivity, and strength. The microstructures of selected samples were also evaluated using scanning electron microscopy. The density was measured for all of the aged specimens.

The normalized thermal conductivity measured at 900°C is plotted as a function of aging time and temperature in Figure 1. At the lowest temperature of 1800°F the thermal conductivity was relatively independent of aging time. At 2100°F, the thermal conductivity increased steadily with aging time and was 1.7 times greater than the as-sprayed value after 5000 h. At the highest aging temperature of 2300°F, the thermal conductivity after 500 h was 2.7 times that of the as-sprayed specimen. For times exceeding 500 h, the thermal conductivity did not change appreciably. As shown in Figure 2, the apparent density also increased with aging time and temperature. As discussed previously, subsequent SEM showed that sintering was the primary cause of these density variations. It is also likely that sintering of the TTBC was a major cause of the increase in thermal conductivity.

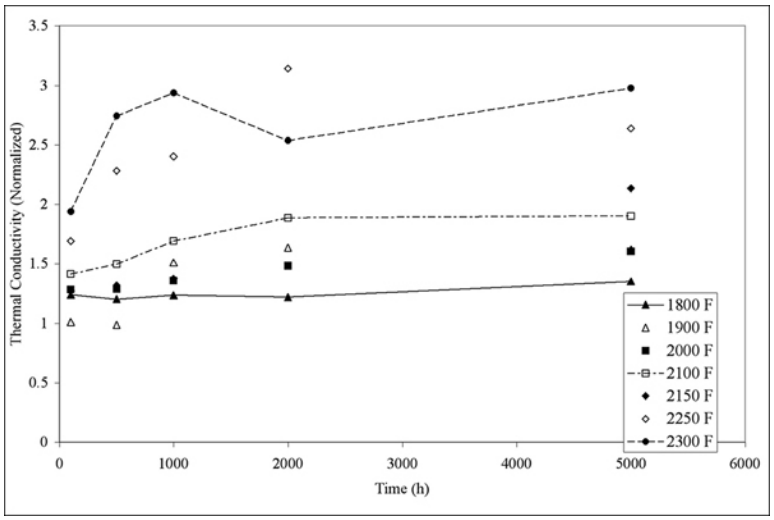

Figure 1: Thermal conductivity (measured at 900°C) as a function of aging temperature and time. The thermal conductivity was normalized by dividing the experimental data by the value measured for the as-sprayed samples.

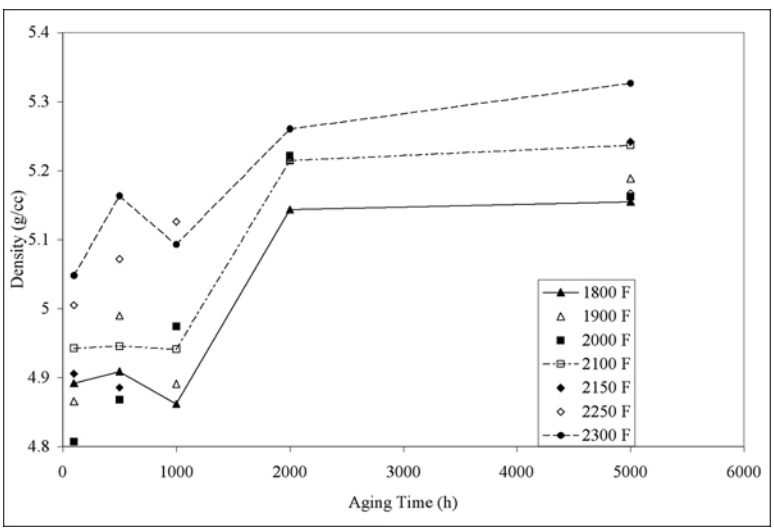

Figure 2: Apparent density as a function of aging temperature and time.

The steam injection system (Figure 3) described in the previous quarterly report was also used to evaluate the temperature sensitivity of recession of sintered alpha silicon carbide. Currently three specimens are being exposed at 1100, 1200, and 1300°C.

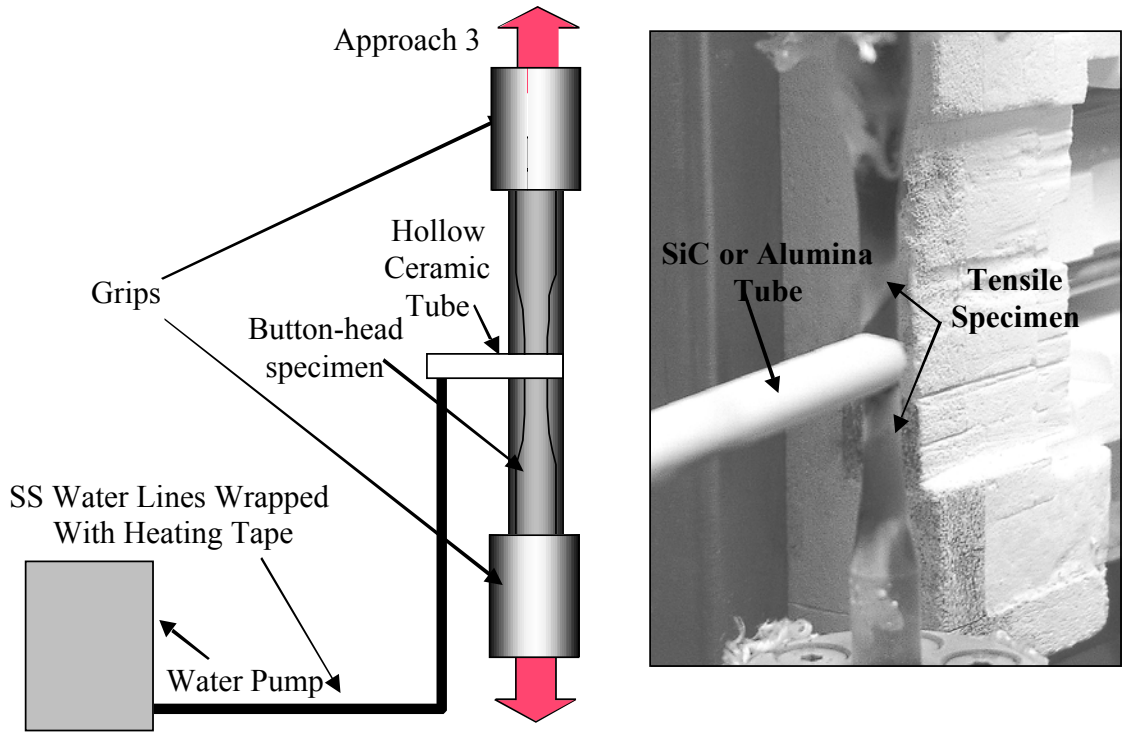

Figure 3: Direct Steam Injection System

Status of Milestones

Milestone 1: Design, fabricate, and evaluate steam containment system for existing creep-stress rupture rigs and issue letter report (April 1, 2001/Delayed 12 months/Completed April 10, 2002).

Milestone 2: Conduct preliminary environmental stability tests on uncoated SN282 and issue letter report (July, 2004/ Delayed 12 months).

Milestone 3: Modify 4 test frames to accommodate direct steam injection system (March 2002/Completed June 2002).

Industry Interactions

Monthly teleconferences were held with UTRC to discuss results of ORNL activities conducted in support of their microturbine program.

Problems Encountered

None

Publications

None

Reliability Analysis of Microturbine Components

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Objective

The objective of this project is to work closely with microturbine manufacturers and suppliers to provide technical expertise relative to component reliability analysis and statistical parameter estimation. This subcontract is separated into two tasks as outlined below:

Task 1 – Develop and enhance parameter estimation software.
Task 2 – Provide design support to companies involved in the DER project.

Task 1 is motivated by the materials needs of the various microturbine manufacturers to evaluate ceramic material provided by St. Gobain, Kennametal and Kyocera. The parameter estimation software is available to all participants in the DER program, and it is now interfaced with the NASA CARES reliability software. The second task is motivated by the need of the microturbine community to have the most up to date methods of analysis available in the design of components fabricated from ceramics.

Highlights

Connecticut Reserve Technologies, LLC (CRT) has worked with Ingersoll Rand (IR) to analyze a CMT gasifier rotor. IR provided CRT with finite element models that captured both transient and steady state thermo-mechanical stress fields. CRT conducted and is in the process of analyzing each load step with the NASA *CARES/Life* reliability algorithm. Current as well as historical silicon nitride failure data (fast fracture) was provided to CRT and Weibull parameters were estimated using CRT's WeibPar, the Weibull parameter estimation algorithm referred to above. CRT conducted a comparative fast fracture evaluation of materials (e.g., AS800, SN282, NT-154) using the NASA CARES/Life reliability algorithm.

Now CRT is developing a methodology to ascertain Weibull distribution metrics for the Ingersoll Rand rotor (see discussion on technical progress in the next section). The combination of service stress states and a requisite probability of survival for a Si3N4 microturbine rotor are being used to determine Weibull modulus - characteristic strength pairs for an arbitrary Si3N4. These data pairs will be provided relative to standard test coupons and methods (e.g., ASTM C1161B and the ORNL button head test specimens). The material characteristic strengths are determined for a range of Weibull moduli, and then are scaled to standard test coupons and test methods. The intent is to provide a methodology to define Weibull distribution metrics for Si3N4 vendors in terms of data generated from standard test specimens. These metrics will be associated with a specified probability of survival for the rotor.

Technical Progress

Task 1 – Progress related to Task 1 is as follows:

- The bootstrap confidence bound methodology for censored failure data was implemented.

- Output to a text file with plotting information – the output file (*.wpr) contains the necessary information (failure data points, estimated line endpoints, etc.) so users may import WeibPar calculations to their own graphics package. CRT has worked closely with Oak Ridge National Laboratory (ORNL) personnel on this option.

- Output to a CARES material parameter file – this file (*.cmp) is compatible as input to CARES/Life version 6.1. This latest version of CARES has been developed by CRT under separate contract for the NASA Glenn Research Center.

- Although incomplete at this time the capability of pooling failure data across multiple temperatures has been studied and is currently being implemented. The methodology for these routines has been used under separate contract for the ARMY Research Laboratory (ARL).

Task 2 –CRT has begun generating results relative to defining material parameters for the Ingersoll Rand rotor. Figure 1 depicts the relationship between the Weibull modulus and the material characteristic strength *(σ_0)* for a stipulated failure rate of 1 in 500,000.

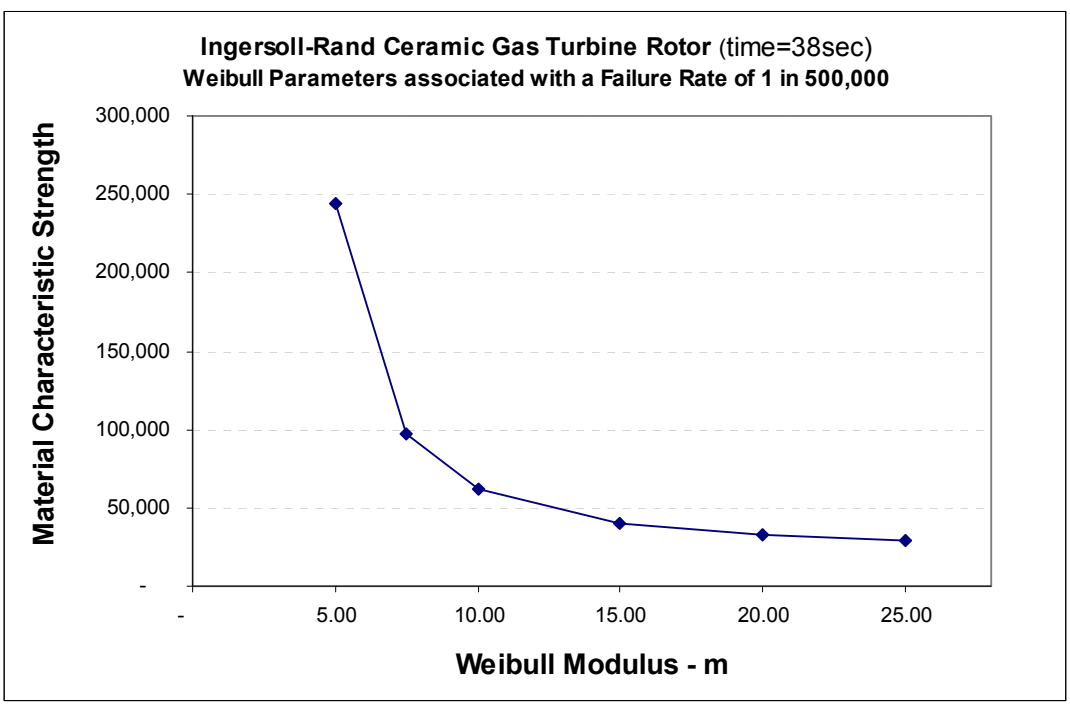

Figure 1 Material Characteristic Strength as a Function of the Weibull Modulus

The information in Figure 1 can be cast in a more relevant context if the relationship between the material characteristic strength and Weibull characteristic strength *(σ_θ)* is utilized. This requires adopting a specific test specimen. In Figure 2 the characteristic strength of an ORNL button head specimen is graphed as a function of the Weibull modulus.

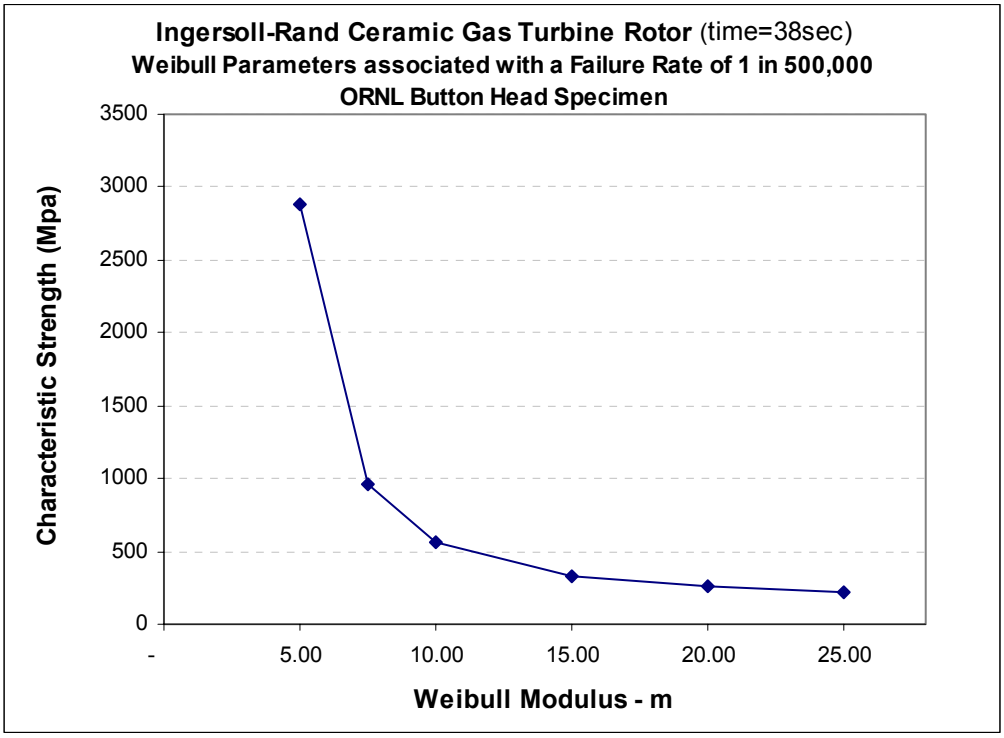

Figure 2 Characteristic Strength of the ORNL Button Head Specimen as a Function of the Weibull modulus.

This is the type of metric discussed above that is currently under development.

Status of Milestones

On schedule

Industry and Research Interactions

Software enhanced under this subcontract is being utilized by the Army Research Laboratory in the development of ceramic gun barrels and ceramic armor.

Problems Encountered
None

Publications/Presentations

1. "Life Prediction of Structural Components," S.F. Duffy, L.A. Janosik, A.A. Wereszczak, B. Schenk, A. Suzuki, J. Lamon, and D.J. Thomas, in Progress in Ceramic Gas Turbine

Development: Volume 2 Ceramic Gas Turbine Component Development and Characterization, M. van Roode, M.K. Ferber, and D.W. Richerson, volume chairs, ASME Press, pp. 553-606, 2003.

2. "Fine Ceramics (Advanced Ceramics, Advanced Technical Ceramics) - Weibull Statistics for Strength Data," S.F. Duffy, ISO Standard Designation: FDIS 20501, (2003).

CHARACTERIZATION OF ADVANCED CERAMICS FOR INDUSTRIAL GAS TURBINE/ MICROTURBINE APPLICATIONS

Oxidation/Corrosion Characterization of Monolithic Si_3N_4 and EBCs

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Objectives

Characterization and corrosion analyses of Si_3N_4 materials provided to ORNL as part of the Hot-Section Materials/Component Development Program

Exposures of candidate Si_3N_4 materials to high water-vapor pressures (in Keiser Rig) to simulate high-temperature, high-pressure environmental effects associated with microturbines

Evaluation of the reliability of environmental barrier coatings (EBCs) on silicon nitrides for selected microturbine applications

Highlights

An atmospheric thermogravimetric (microbalance) rig is being used to conduct initial screening of possible EBC compositions. Preliminary thermogravimetric work with barium-strontium aluminosilicate (BSAS) and calcium aluminate showed fundamentally good stability in the presence of water vapor as evaluated in this manner.

Technical Progress

An atmospheric thermogravimetric (microbalance) facility at ORNL is being used for initial screening of candidate EBC compositions in conjunction with parallel efforts on coating development. In particular, initial work is in collaboration with the work of Armstrong et al.[1] and is focused on the basic oxidation response of bulk forms of possible EBC compositions in the presence of water vapor. While the complete effects of water vapor on oxidation of materials in combustion environments are not manifested at the low partial pressures associated with rigs operated at low gas-flow rates or at atmospheric pressure, the use of ambient-pressure thermogravimetry can help discriminate between different candidate EBC compositions.[2] Bulk specimens of possible coating compositions are used in order to investigate fundamental chemical compatibility issues rather than those associated with coating integrity or adherence. Obviously, those compositions that do not show good oxidation resistance under these test conditions are not candidates for further development as EBCs.

The initial set of EBC-related experiments in the thermogravimetric facility involved bulk specimens of BSAS prepared at ORNL by B.L. Armstrong. The specimens were first uniaxially pressed followed by cold isostatic pressing to increase pack densities and heat treatment in a box furnace. The BSAS composition is considered current state-of-the-art EBC for combustion

applications around 1200°C, but other EBCs will be needed for extended life at this temperature and where significantly higher temperature operation is needed.[2,3] This system therefore can be used as a benchmark for other developmental EBC compositions. While suspended from a microbalance, individual BSAS specimens (approximately 27x18x5 mm) were exposed at 1200°C in a slowly flowing (~100 cc/min) gas of dry air or air-20%H_2O near 1 atm. Mass was thus continuously recorded as a function of oxidation time as the specimen was held isothermally in an oxidizing environment. The resulting specimen mass change at a particular time, Δm, can be approximated as

$$\Delta m = m_o - m_v \tag{1}$$

where m_o is the amount of oxygen uptake by oxidation and m_v is the mass of any volatile product that may form.

The first thermogravimetric results for BSAS are shown in Fig. 1. The BSAS showed very low reaction rates in both air and air-20%H_2O with net mass gains in both cases. The measurements have been corrected for evaporation of the Pt wire used to secure the specimens, but because of the very small differences in mass being measured, the magnitude (and sign) of the mass changes are very sensitive to the accuracy of the correction factor. However, this does not affect any relative differences observed as compositions and environments are changed. Regardless, the

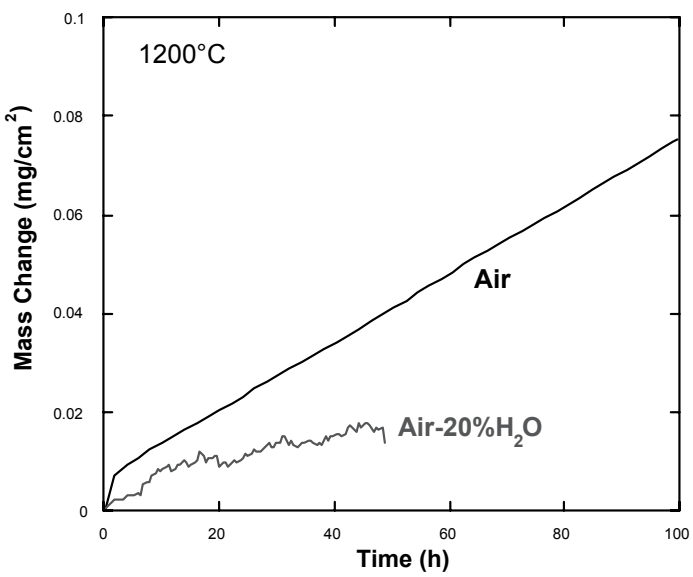

Figure 1. Mass change as a function of time for BSAS exposed at 1200°C at 1 atm.

present results indicate fundamentally good stability of the BSAS under these environmental conditions.

As the measured Δm is the net result of reactions that lead to O_2 uptake and material loss (see eq. 1), the smaller mass gains measured for the BSAS exposed at an elevated H_2O pressure may indicate a susceptibility to volatility reactions in the presence of water vapor. The production of volatile species from oxidation of BSAS in not unexpected: the formation of $Si(OH)_4$ is well documented,[4] the oxides of Ba and Sr have fairly high vapor pressures,[5] and some material loss of a BSAS EBC has been observed in an engine test.[3] The important point in the present context is that thermogravimetric analysis at low gas flow velocities can provide an indicator of overall

oxidation susceptibility. However, more work is needed to check reproducibility and confirm this observation for various standard EBC materials and SiO_2 because the mass changes being measured are very small and close to the resolution limits of the balance.

The $CaO\text{-}Al_2O_3$ system is also being investigated as an oxidation/corrosion-resistant coating for various applications. The initial result for a bulk calcium aluminate exposed in the thermogravimetric system is shown in Fig. 2, which also contains the BSAS data of Fig. 1 for comparison. The data indicate fairly good stability of the calcium aluminate in the 20% H_2O environment at 1200°C.

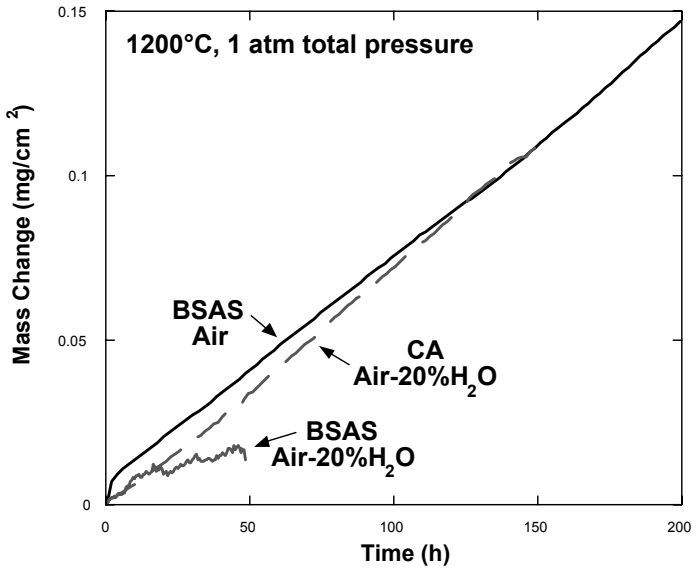

Figure 2. Mass change as a function of time for BSAS and $CaO\text{-}Al_2O_3$ (CA) exposed at 1200°C at 1 atm.

Status of Milestones

08/2003 Report results of initial evaluation of "new" Si_3N_4 materials from Saint-Gobain and Kennametal exposed in the Keiser Rig for two temperatures and two water-vapor pressures.
Two Si_3N_4 materials have been received from both Kennametal and St. Gobain and are currently being exposed in the Keiser Rig at 2 different water-vapor pressures.

Industry Interactions

Conference call with United Technologies Research Center to discuss EBC development and collaborative efforts, September 17, 2003.

Problems Encountered

None

Publications/Presentations

P. F. Tortorelli and K. L. More, "Effects of High Water-Vapor Pressure on the Oxidation of SiC at 1200°C," *J. Am. Ceram. Soc.* 86 (2003) 1255-62.

K. L. More, P.F. Tortorelli, L.R. Walker, N. Miriyala, J.R. Price, and M. van Roode, "The High-Temperature Stability of SiC-Based Composites in High Water-Vapor Pressure Environments," *J. Am. Ceram. Soc.* 86 (2003) 1272-81.

References

[1] See, for example, B.L. Armstrong, M.P. Brady, K.M. Cooley, J.A. Haynes, and H.T. Lin, "Environmental Protection Systems for Ceramics in Microturbines and Industrial Gas Turbine Applications, Part B: Slurry Coatings and Surface Alloying," DER Materials Quarterly Progress Report for Period Ending June 30, 2003, www.eere.energy.gov/der/tech_base/tech_base.html#advmatl.

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Mechanical Characterization of Monolithic Silicon Nitride Si_3N_4

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Objective

The objective of this project is to work closely with microturbine materials suppliers to characterize monolithic ceramics and provide the data obtained to microturbine manufacturers via a Web site database.User-friendly software, that will allow prospective users to readily compare different silicon nitrides, will also be developed. This project consists of the following four tasks:

- Task 1: Evaluate Strength and Slow Crack Growth of New Materials
- Task 2: Modify Six Existing Creep Frames to Allow Introduction of Water Vapor
- Task 3: Evaluate the Effects of Water Vapor Upon Honeywell's Silicon Nitride Ceramics
- Task 4: Develop "User Friendly" Software for Searching Existing Mechanical Properties Database

Task 1 is motivated by the materials needs of Ingersoll-Rand (IR) Energy Systems, General Electric, and UTRC. The ceramic materials being considered by IR Energy Systems include Kyocera's SN-235 and SN-237 for which the required mechanical property data are somewhat limited. In the case of the UTRC microturbine, Si-SiC is a prime candidate for the combustor. Consequently, Task 1 will focus on the generation of key mechanical property data for these materials with emphasis on strength (and strength distribution), time dependent failure (evaluated using dynamic fatigue), elastic properties, and fracture toughness.

In FY 2003, baseline mechanical property data will be measured for materials supplied by Kennametal and St. Gobain Advanced Ceramics as part of their materials development effort funded by ORNL. The initial focus will be on measuring flexure strength as a function of temperature and stressing rate.

In Task 2, two approaches for environmental testing will be considered. The first method, which was previously used on two test frames, involves the uniform injection of steam into the furnace cavity. The primary advantage of this arrangement is that extensometry can still be used to evaluate creep deformation. The second approach utilizes a small ceramic tube to inject water directly onto the gage section of the buttonhead specimen. This approach will be evaluated in FY2003 using SN-281 buttonhead specimens.

Task 3 is currently on hold due to the unavailability of material.

In FY 2003 in Task 4 the data sets generated at ORNL will be added to the UDRI Web site.

Highlights

The database Web site has been modified and input files are being constructed for the CARES computer design code. Testing of Kyocera's SN281 silicon nitride at 1400 °C and 1500 °C in air showed that the stress exponent was constant (n=24) at these temperatures. This is in contrast to most silicon nitrides that show decreasing n values with increasing temperature.

Technical Progress

Testing of Kyocera SN-281 and SN-282 Silicon Nitride

Ten billets of SN281 were received from Dr. Ferber at ORNL. Six billets were sent to Chand Kare for machining into size B flexure specimens. Dynamic fatigue testing in air at 1400 °C and 1500°C has been completed this quarter by stressing test bars at both 0.003MPa/s and 30MPa/s . All samples exhibited plastic deformation with every failure occurring at a surface flaw.

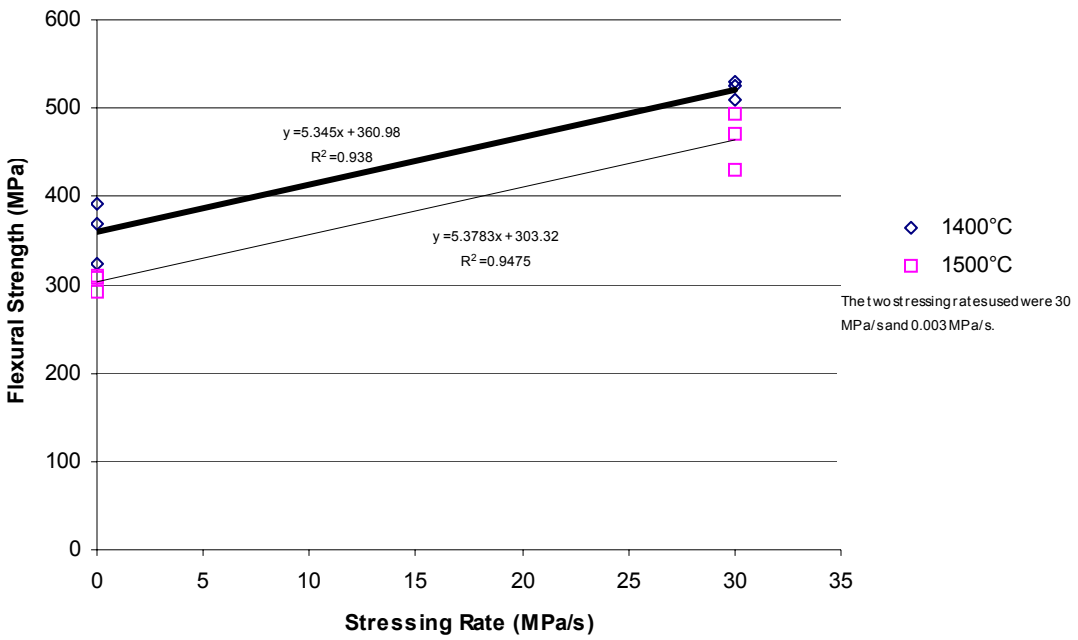

Figure 1. Dynamic fatigue of SN281 in air at 1400°C and 1500°C (n=24).

Figure 1 shows a least means squares graph of strength versus stressing rate at two temperatures in air. This was done to see if there was any discernable difference in the values of the stressing exponent value (n) at these two temperatures. This was calculated to be 24 for both temperatures. This is in contrast to most silicon nitrides that show the value of n to be decreasing as a function of temperature. The classic explanation of slow crack growth invokes crack formation ahead of the main crack as a result of grain boundary sliding. Since the viscosity of the grain boundary phase decreases with increasing temperature the value of n increases. This mechanism does not appear to be operating in SN281 silicon nitride. Rather there appears to be some threshold based mechanism since an increase in temperature does not cause enhanced slow crack growth.

Upgrading the Website

The Web site has been modified so that users can obtain design input files based upon the property database. These Excel files are shown as .xls files, and can be used as input files for designing ceramic components using such programs as NASA's CARES computer code. Most of the data for the creep properties and strength of SN88 and AS800 has been collected in this format so far. This includes test conditions, specimen geometry, surface finish and failure stresses. Files for the other materials in the property database will be constructed in the coming year. The modified website can be viewed at

http://www.udri.udayton.edu/design_data_files

Aluminum Silicon Carbides: A Potential EBC

Aluminum containing ceramics generally possess good oxidation resistance and are thus worthy of consideration as EBCs. While many of these have been considered or are currently under investigation as potential EBCs, one group of aluminum containing ceramics that has received little attention to date is the aluminum silicon carbides. Although little property data exists for these materials, initial oxidation studies on Al_4SiC_4 (1) indicate the formation of a thin protective oxide layer of alumina and mullite. The objective of the current task is to determine the oxidation resistance of these materials in water vapor and to determine some of their basic properties and hence ascertain whether or not these materials can be considered as viable EBC materials.

The aluminum silicon carbides found to date (Al_4SiC_4, $2Al_4C_3.SiC$ and $Al_4C_3.SiC.AlN$) have similar structures and an aluminum content of about 45 atomic weight. Therefore it seems probable that the oxidation resistance of these three compounds would be similar. Efforts to make the two compounds $2Al_4C_3.SiC$ and $Al_4C_3.SiC.AlN$ were thus terminated.

Analysis of several hot pressings showed that at 1750 °C there is still a small amount of unreacted Al_4C_3 with the Al_4SiC_4, whereas at 1900 °C and above free aluminum and sometimes carbon is seen. As a result an additional hot pressing at 1825°C was performed. This produced largely Al_4SiC_4 with a small amount of $Al_4Si_2C_5$. This result is in agreement with the results shown in Figure 1 of Oscroft and Thompson's paper (2).

Equipment is currently being setup to examine the oxidation of Al_4SiC_4 in air and air/water vapor and to compare it with that of hexalloy silicon carbide and SN281 silicon nitride.

Status of Milestones

(a) Testing and Evaluation of SN-281/282

Additional samples have been obtained in order to evaluate this grade of silicon nitride at 1400 °C and 1500 °C, and to determine its oxidation and slow crack growth behavior in air and water vapor.

Industry and Researcher Interactions

Discussions were briefly made with John Holowczak of UTRC over current challenges for developing EBCs.

Problems Encountered

None

Publications

None

References

1. K. Inoue, A.Yamaguchi, and S.Hashimoto, "Fabrication and Oxidation Resistance of Al_4SiC_4 Body", *J.Ceram Soc. Jpn*. 110[11], 1006-1011, 2002.

2. Robert J. Oscroft and Derek P. Thompson, *J. Amer. Ceram. Soc*. 75 (1), 224-226 (1992).

Microstructural Characterization of CFCCs and Protective Coatings

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Objectives

Characterization of CFCC materials and CFCC combustor liners after exposure to simulated (ORNL's Keiser Rig) and actual (engine tests) combustion environments

Exposures of candidate environmental barrier coatings (EBCs) to high water-vapor pressures (in Keiser Rig) to determine thermal stability and protective capability

Work with CFCC and coating suppliers/manufacturers to evaluate new/improved ceramic fibers, protective coatings, and composite materials

Highlights

During this quarter, additional work was initiated with ATK-COI Ceramics, Inc. to further characterize Alumina/Nextel 720 (A/N720) oxide/oxide continuous fiber ceramic composite coupons at high water-vapor pressures in ORNL's Keiser Rig at both 1200°C and 1250°C. As reported previously (DER Quarterly Progress Reports for 07/02-09/02, 10/02-12/02, 01/03-03-03, and 04/03-06/03), this same A/N720 composite material was exposed for up to 3000 h at 1135°C, 10 atm total pressure, in a N_2-10%O_2-10%H_2O-6%CO_2 gas mixture used to simulate the combustion environment of a Capstone microturbine. The material showed no loss of mechanical properties or changes in microstructure after long-term exposures at 1135°C.

Technical Progress

In order to determine the maximum use temperature for A/N720 oxide/oxide CFCCs, exposures similar to those conducted at 1135°C will also be conducted at the higher temperatures of 1200°C and 1250°C. Post-exposure analyses, including mechanical and microstructural evaluation, will be conducted after 1000, 2000, and 3000 h exposures in the Keiser Rig. No changes in structure or properties were observed after the A/N720 was exposed for 3000 h at 1135°C in the N_2-10%O_2-10%H_2O-6%CO_2 gas mixture (DER Quarterly Progress Report 01/03-03/03). The first 1000 h exposure at 1200°C is near completion.

In addition to the Keiser Rig exposures, smaller coupons of the A/N720 CFCC are currently being exposed for 500 h at 1200°C at 1 atm in flowing dry air or air + 10 vol% H_2O in a microbalance so that stability over an extended period of time can be monitored gravimetrically. Similar experiments were also conducted at 1135°C (DER Quarterly Progress Report 10/02-12/02) and will also be conducted at 1250°C. In this way, any direct effect of water vapor at the different exposure temperatures on microstructure and/or mechanical properties can be determined by comparing the data.

Status of Milestones

03/02 Milestone — Complete a comprehensive report on oxidative degradation mechanisms of Si-based ceramics and rates of such materials at high temperatures and high water-vapor pressures typical of gas turbines.

This milestone has been completed. A manuscript has been published by P. F. Tortorelli and K. L. More, "Effects of High Water-Vapor Pressure on Oxidation of SiC at 1200°C," *Journal of the American Ceramic Society*, **86**[8] 1249-55 (2003).

04/02 Milestone — Prepare a report and present results on the evaluation of the set of 14,000h Texaco (Chevron) combustor liners.

This milestone has been completed. A manuscript has been published by ASME and is part of the proceedings of the IGTI Turbo Expo 2002 in Amsterdam, June 3-6, 2002. K. L. More, P.F. Tortorelli, L.R. Walker, J.B. Kimmel, N. Miriyala, J.R. Price, H.E. Eaton, E.Y. Sun, and G.D. Linsey, "Evaluating EBCs on Ceramic Matrix Composites After Engine and Laboratory Exposures," ASME Paper #GT-2002-30630.

ASME Paper #GT-2002-30630 was awarded 2002 Best Paper by the Ceramics Committee of IGTI as well as being selected for publication in Journal of Engineering for Gas Turbines and Power.

04/03 Milestone — Prepare a report and present results on the evaluation of the set of 15,000 h Malden Mills engine-tested combustor liners.

This milestone has been completed. Results from the study were presented at a closed meeting with Solar Turbines, United Technologies Research Center, Argonne National Laboratory, and ORNL during the 27th Annual Ceramics and Composites Conference in Cocoa Beach, FL on January 29, 2003. A manuscript was prepared by Solar Turbines,

Inc., which summarizes these results. ASME Paper #GT-2003-38920, "The Evaluation of CFCC Liners After Field Testing in a Gas Turbine – IV," by J. Kimmel, J.R. Price, K.L. More, P.F. Tortorelli, E.Y. Sun, and G.D. Linsey, was presented at IGTI Turbo Expo 2003 in Atlanta, GA, June 15-19, 2003.

09/03 Milestone — Complete first-stage evaluation of the effects of water vapor on the oxidation of at least two new candidate compositions for protective coatings.

This milestone has been completed. EBCs supplied by UTRC, Honeywell Ceramic Components, Northwestern University, SMAHT Ceramics, Inc., and University of Colorado have been exposed in the Keiser Rig. Results from these exposures have been reported to the individual coating producers. Some results were published in paper by K.L. More, P.F. Tortorelli, and L.R. Walker, "Verification of an EBC's Protective Capability by First-Stage Evaluation in a High-Temperature, High-Pressure Furnace," ASME Paper #GT-2003-38923

Industry Interactions

Worked with Andy Szweda of ATK-COI Ceramics, Inc. to acquire A/N720 CFCC material and determine exposure conditions for Keiser Rig runs.

Worked with Rama Nageswaran of SMAHT Ceramics, Inc. on the exposure of candidate EBC compositions in the Keiser Rig.

Problems Encountered

None

Publications/Presentations

1. K. L. More and P. F. Tortorelli, "Evaluation of EBCs in ORNL's Keiser Rig," presented at the EBC Workshop in Nashville, TN, November 6-7, 2002.

2. K. L. More, P. F. Tortorelli, L. R. Walker, H. E. Eaton, E. Y. Sun, G. D. Linsey, J. B. Kimmel, N. Miriyala, and J. R. Price, "Evaluating EBCs on Ceramic Matrix Composites After Engine and Laboratory Exposures," presented at IGTI Turbo Expo 2002, Amsterdam, June 3-6, 2002. ASME Paper #GT-2002-30630. Selected

for publication in *Journal Engineering for Gas Turbines and Power*. This paper won the Best Paper Award for Ceramics Division.

3. K. L. More and P. F. Tortorelli, "The High-Temperature Stability of SiC-Based Composites in High Water-Vapor Pressure Environments," *Journal of The American Ceramic Society*, **86**[8] 1272-81 (2003).

4. P. F. Tortorelli and K. L. More, "Effects of High Water-Vapor Pressure on Oxidation of SiC at 1200°C," *Journal of The American Ceramic Society*, **86**[8] 1249-55 (2003).

5. K. L. More, P. F. Tortorelli, and L. R. Walker, "Verification of an EBC's Protective Capability by First-Stage Evaluation in a High-Temperature, Hig-Pressure Furnace," ASME Paper #GT-2003-38923. Presented at IGTI Turbo Expo 2003 in Atlanta, GA, June 16-19, 2003

6. J. B. Kimmel, J. R. Price, K. L. More, P. F. Tortorelli, E. Y. Sun, and G. D. Linsey, "The Evaluation of CFCC Liners After Field Testing in a Gas Turbine – IV," ASME Paper #GT-2003-38920. Presented at IGTI Turbo Expo 2003 in Atlanta, GA, June 16-19, 2003.