DE MATERIALS QUARTERLY PROGRESS REPORT

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October—December 2004

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RECUPERATORS

Recuperator Alloys – Composition Optimization for Corrosion Resistance

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Objective

In order to provide a clear, fundamental understanding of alloy composition effects on corrosion resistance of stainless steel components used in microturbine recuperators, the oxidation behavior of model and commercial alloys is being studied. Low alloy steels exhibit accelerated corrosion attack caused by water vapor in exhaust gas at 650°-800°C. An improved mechanistic understanding will improve life-prediction models and will assist in the selection and/or development of cost-effective alloys for recuperators. Issues that continue to be investigated include the effects of temperature, alloy grain size, phase composition and minor alloy additions.

Highlights

Initial microprobe (EPMA) characterization was completed on a series of commercial alloy foils, NF709, HR120 and 625, oxidized for 10,000h in humid air at 650°C and 700°C. All of the foils showed selective grain boundary depletion of Cr near the surface which leads to Fe-rich nodule formation in NF709 and HR120 after exposure at 650°C.

Technical Progress

Multiple specimens of new commercially made foil versions of HR120 and AL20/25+Nb from Allegheny Ludlum (AL) are being tested in standardized exposures using 100h cycles at 650°, 700° and 800°C in air + 10%H_2O. Specimens will be periodically removed from the test for characterization. New laboratory-scale versions of Fe-20Cr-20Ni have been cast and will be rolled to sheet and foil thickness for creep and corrosion testing.

Based on the EPMA results of Cr depletion in commercial foils, a clearer understanding of the role of water vapor has been developed. At 800°C, the Cr depletion is more uniform in the foil cross-section due to rapid Cr diffusion in the metal. However, at 650°C, the depletion is localized to only 12-15μm into the metal and the grain boundaries are selectively depleted due to the slower diffusion of Cr in the bulk alloy. In localized areas of Cr depletion, Fe-rich oxide nodule formation was observed on both NF709 and HR120 after 10,000h at 650°C. However, the material at the center of the foil was relatively unaffected, showing no change in bulk composition. The results after 10,000h indicated a similar rate of Cr depletion from NF709, HR120 and alloy 625 at both 650° and 700°C.

Status of Milestones

Submit an open literature publication with long-term data supporting the development of low cost, oxidation resistant stainless steels for exhaust gas environments. (July 2005, on track)

Industry Interactions

Discussed alloy development progress with J. Rakowski of AL at ASM meeting, Columbus, OH, in October and received new AL20/25+Nb material from AL through P. Maziasz at ORNL.

Recuperator Materials Testing and Evaluation

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Objective

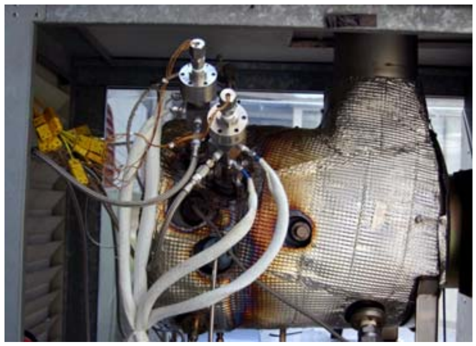
The objective of this sub-task is to screen and evaluate candidate materials for the next generation of advanced microturbine recuperators. To attain this objective a microturbine was modified to operate at recuperator inlet temperatures as high as 850°C. The durability of candidate recuperator materials is determined by placing metallic foil test specimens at a location upstream of the recuperator, followed by determination of the evolution of the material's physical and mechanical properties as a function of time of exposure. Metallic foil test specimens can be subjected to various levels of mechanical stress by pressurizing a specially designed sample holder onto which the metallic foil test specimens are welded. The selection of materials for evaluation is determined in collaboration with other tasks in this program and with manufacturers of microturbine recuperators.

Highlights

Foils of ORNL-modified stainless steels were found to retain 90% of their tensile strength after a 500-hr exposure in ORNL's microturbine recuperator testing facility at 700°C.

Technical Progress

During the reporting period the following alloys were evaluated in ORNL's microturbine recuperator testing facility: four ORNL-modified stainless steels, alloy 625, Haynes alloys 120® and 230®, and FeCrAl. The mechanical properties and microstructure of metallic foil test specimens were evaluated after test exposures of 500 and 1000 hours. It was found that 100-μm thick foils of an ORNL-modified stainless steel containing 19.3% Cr and 12.6% Ni retained 90% of their as-processed tensile strength after a 500-hr exposure at 700°C and that they did not experience degradation of strength and ductility at 653°C. While the 0.2% yield strength of these materials remained unchanged after a 500-hr exposure at 700°C, it increased by 40% at 653°C. While 200-μm thick foils of alloy LCF-625 were found to retain their ultimate tensile strength after a 500-hr exposure at 740°C, they experienced a 30% loss in ductility. 50-μm thick foil test specimens of FeCrAl (15% Cr and 6% Al) experienced significant creep deformation and ruptured during the test. Ongoing work is focused on characterizing the evolution of the foils' microstructure after exposure.



Status of Milestones

1) Complete 1000-hr test campaigns and characterization of HR-120® and ORNL-modified stainless steels (December 2004). Completed.
2) Publish a technical paper that summarizes the testing of HR 120 and NF 709 in ORNL's microturbine recuperator test facility (June 2005). On Track.

Industry Interactions

Continued collaborating with Solar Turbines (San Diego, CA) on the evaluation of the creep resistance of alloy 625 and various grades of stainless steels. Received FeCrAlY foils from Engineered Materials Solutions (Attleboro, MA) for evaluation in ORNL'S microturbine recuperator testing facility.

Advanced Alloys for High Temperature Recuperators

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Objective

The main objective is for ORNL to work with recuperator OEM's and commercial foil and sheet suppliers to test, evaluate and enable the manufacture of recuperators with alloys that have improved temperature capability and performance at a reasonable cost. The near term goal is meeting reliability goals of 40,000-80,000h at about 700^{o}C, while the longer term goal is performance up to 750^{o}C or higher, without sacrificing lifetime. Last year, ORNL began a collaborative project with Allegheny-Ludlum to produce a wide range of commercial sheets and foils of the new AL20-25+Nb stainless alloy for properties characterization testing and recuperator manufacturing trials. Phase I of that project is nearly complete, and Phase II began in FY2005 to study processing parameters to determine the effects of microstructural condition for optimum creep-resistance for the various sheet and foil products. The goal this year is to characterize properties and performance of recuperator air-cells manufactured from AL20-25+Nb alloy.

Highlights

Allegheny-Ludlum has completed processing and delivery of various sheets and foils of AL20-25+Nb alloy to recuperator OEM's. ORNL is completing creep-rupture testing and microstructural characterization. Allegheny-Ludlum has begun processing of Phase II foils and sheets.

Technical Progress

Recuperators made from 347 steel have recently been found to suffer severe moisture-induce oxidation attack and mechanical deformation and failure if moisture and stress exceed critical levels at temperatures above 650^{o}C. Properties characterization and recuperator trials demonstrate that the Ni-based superalloy 625 has sufficient oxidation and creep-resistance for use to about or slightly above 700^{o}C, but at 3.5-4 times the cost of stainless steel, it may not be cost effective for all applications. Austenitic stainless alloys HR120 and AL20-25+Nb have also been identified as more cost effective higher performance alternatives to 347 stainless steels for recuperator applications.

Last year, ORNL began a collaborative program with Allegheny-Ludlum to produce a wide range of commercial sheets and foils of the new AL20-25+Nb alloy appropriate for recuperator air-cell manufacturing. Phase I of this project took standard processing of this alloy and produced materials with significantly better creep-rupture resistance at 700-750^{o}C relative to 347 steel. Phase I material has been delivered to recuperator OEMs for manufacturing trials. ORNL and Allegheny-Ludlum have defined a Phase II effort to examine processing/microstructure effects on optimizing the creep-resistance of various sheets and foils. Phase II foil and sheet production will be completed next quarter, and ORNL properties and microcharacterization will begin.

Status of Milestones

1. Complete mechanical testing and microstructural characterization of various foils and sheets of new AL20-25+Nb alloy, and evaluate the effects of modified processing on creep resistance. March 2005 – on track

Industry Interactions

1. ORNL communicates regularly with Allegheny-Ludlum Technical Center (Chuck Stinner) to provide input, get feedback and monitor progress of this project.

CERAMIC RELIABILITY FOR
MICROTURBINE HOT-SECTION COMPONENTS

Reliability Evaluation of Microturbine Components

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Objective

The objective of this study is to facilitate the successful implementation of complex-shaped ceramic components in advanced microturbines to significantly increase efficiency and reduce NOx emission. This work also provides a critical insight into how the microturbine environments influence the microstructure and chemistry, thus mechanical performance of materials.

Highlights

High temperature steam jet evaluation of an experimental multilayered EBC system based on $Lu_2Si_2O_7$ and $Lu_2Si_2O_7$/Mullite eutectic and developed by AIST, Japan at 1300°C for 500h has been completed.

Technical Progress

SEM analysis on NT154 silicon nitride samples showed that a substantial amount of un-crystallized oxynitride glass was observed at triple grain boundaries of the as-received bulk material. The oxynitride glassy phase softens at temperatures ≥1000°C, evident by the viscous ligament and flow feature, and could lead to the onset of slow crack growth and creep process, resulting in low fatigue exponents measured. Thus, the processing routes plus the sintering cycle need to be modified in order to completely eliminate the oxynitride glass.

The machining of NT154 silicon nitride MOR bend bars manufactured with diffusion barrier coating (DBC) has been significantly delayed due to personnel issue at the PremaTech Chand.

High temperature steam jet tests have been carried out on RE-disilicates fabricated by AIST, Japan at temperature of 1300, 1400, and 1500°C for 100h. The objective of this study is to provide the recession and phase stability as a function of temperature. Also, the activation energy for the above mentioned process could be obtained.

Status of Milestones

1. Complete mechanical characterization of NT154 microturbine rotors with and without EBC manufacture under optimized processing conditions. Sept 2005 – on track.

Industry Interactions

1. Communication with Vimal Pujari and Ara Vartabedian at Saint-Gobain on the fractography and SEM results of NT154 silicon nitride samples manufactured under Phase I effort.
2. Communication with Russ Yeckley at Kennametal on the high temperature dynamic fatigue test results of SiAlON ceramics manufactured during Phase I effort.
3. Communication with John Holowczak at UTRC to on the dynamic fatigue results of Saint-Gobain NT154 silicon nitride.

4. Communication with Tatsuki Ohji and Shunkichi Ueno at AIST, Japan on the progress of the steam jet testing of RE-disilicates.

Modeling of Advanced Materials for Microturbine Applications

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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 DE 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. A secondary objective of the program is to develop and characterize the toughened silicon nitride ceramics

Highlights

The Integrated Reliability Assessment Software (described in a previous highlight) was modified to provide input files for CARES (Ceramics Analysis and Reliability Evaluation of Structures).

Technical Progress

A comprehensive database of mechanical properties a number of commercial silicon carbides and silicon nitrides was incorporated into a newly developed software package for implementing reliability analysis. The Integrated Reliability Assessment Software allows users to rapidly implement reliability estimates comparing a number of candidate materials. The results of each analysis are displayed graphically by generating strength/stress versus temperature plots to compare finite element results for a specific component with the strength-temperature data for a number of commercial ceramics from the database. The strength is adjusted to account for both slow crack growth and creep. During this reporting period, the software was modified to provide input files for CARES (Ceramics Analysis and Reliability Evaluation of Structures).

Status of Milestones

(1) Report on the development of integrated reliability assessment software for advanced silicon-nitride. November 2004- Complete

Industry Interactions

1. Discussions were held with John Holowczak and William Tredway, from UTRC; Bill Ellingson form ANL;and Santosh Y. Limaye of Vesta Ceramics, LLC at the EBC Workshop concerning possible collaborations.
2. Communications with Steve Duffy of CRT concerning use of the Integrated Reliability Assessment Software.
3. Discussions were held with Ara Vartabedian from Saint-Gobain Ceramics & Plastics concerning incorporation of current NT154 data into database.

Reliability Analysis of Microturbine Components

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Objective

Develop characteristic strength – Weibull modulus "maps" for both surface and volume distributed flaws that limit strength in ASTM C 1161B and ASTM 1161C bend bars and ASTM C1273 button head tensile specimen geometries. In addition, update the CARES algorithm and update the WeibPar algorithm and make these software algorithms available to DE participants.

Highlights

A method of generating material specific and specimen specific strength characterization maps was developed and applied to the case study of the a complex-shaped gas turbine rotor. The WeibPar and CARES algorithms were updated to automate the generation of these characterization maps. Additionally, component reliability confidence bounds via the bootstrap technique were added to WeibPar and CARES

Technical Progress

CRT has implemented a method that establishes Weibull distribution metrics for silicon nitride vendors based on a relevant component. Service stress states from the various treated surfaces of a rotor blade must be combined with a stipulated component reliability in order to develop material performance curves. These curves must be scaled to standard ceramic test coupons (e.g., bend bar specimens) making component requirements transparent to material vendors.

This allows changes in designs to take place in parallel with enhancements in material properties. Material characterization maps can be generated for a given component under specified operating conditions.

Concurrent efforts to update CARES and WeibPar (e.g. confidence bounds on components, pooled censored data, etc.) will augment the software tools used to generate the maps described above.

Status of Milestones

Create a capability within the CARES algorithm to compute (unitless) area or volume (or both) loading factors (kA and kV, respectively) as a function of Weibull modulus (m). December. 2004 – on track

Industry Interactions

Presented a poster entitled "NDE Technologies for Condition Monitoring of EBCs" at the EBC Workshop on November 17-18, 2005, in Nashville, TN.

NDE Technology Development for Microturbines

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Objective

The objective of this work is to develop reliable, low-cost, non-contact, noninvasive methods that reduce engine risk when new high temperature materials are inserted into advanced, low-emission microturbines and mid-sized gas turbines. It has been demonstrated that to achieve the desired 42% energy efficiency for advanced microturbines, new high temperature materials such as ceramics with environmental barrier coatings are necessary. Further, to achieve the desired low-emissions for mid-sized gas turbine engines, it is necessary to utilize advanced ceramic composite materials with new environmental barrier coatings. Insertion of these new materials into complex engines always carries a risk and methods that can assess the condition of these materials before insertion and after various run times play an important role.

Highlights

1—The new nondestructive method under development for assessing condition of the environmental barrier coatings was further extended with a new higher power laser allowing penetration of thicknesses over 200um. 2—Quantification of the sizes of features measured in the digital images obtained of the ceramic components was advanced through use of a new algorithm. The ability to quantify the dimensions of objects, thus has been greatly improved. 3—Cooperative work with DLR in Cologne, Germany, was started relative to quantification of "defects" in various coating systems .4---Argonne participated in the Oak Ridge Organized Environmental Barrier Coating Workshop held November 18-19, 2004.

Technical Progress

Advances in the OCT system now allows measurement of coating thicknesses of over 200um. This method, based on laser interferometry, allows totally nondestructive measurement of the thickness in local regions. As larger monolithic ceramic components are being developed for insertion into engines, there is more of an issue with dimensional control. If a method can be developed that allows the establishment of these dimensions, both internal as well as external, then this would advance the technology. In this regard, we have established a new algorithm that allows quite exact determination of the edges from 3D x-ray CT scan data sets. In this work, collaboration with others is essential. This period cooperative work was started with DLR (The German Aerospace Institute) in Cologne, Germany. They are preparing special test samples.

Status of Milestones

1. Demonstrate automated feature detection in 3D x-ray topographic images for monolithic ceramics July 15, 2005, – on track
2. Complete conduct of NDE tests on 2 sets of ceramic composite liners run in Solar Turbines field test engines. June 15, 2005, – on track.

Industry Interactions

1. Direct discussions were held with staff of Saint Gobain Industrial Ceramics and Plastics regarding determination of as-cast surfaces in silicon nitride ceramics.
2. Discussions were held with Dr. Matt Ferber of Oak Ridge regarding the coupling of some of his modeling efforts with the NDE data being obtained at Argonne.

CHARACTERIZATION OF ADVANCED CERAMICS FOR INDUSTRIAL GAS TURBINE/ MICROTURBINE APPLICATIONS

Oxidation/Corrosion Characterization of Microturbine Materials

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Objective

Environmental barrier coatings (EBCs) will be required on surfaces of Si-based ceramic and composite materials exposed to microturbine combustion (high water-vapor) environments. Numerous EBC systems are being developed for use on Si_3N_4 hot-section microturbine components. The reliability of these different EBC compositions, in terms of thermal stability, H_2O permeability, and volatility, at high temperature and water-vapor pressures, is being evaluated long-term in the ORNL Keiser Rigs.

Highlights

The initial exposure of several (Ba,Sr)-based aluminosilicates and Si-based standards to extremely high water-vapor pressures in the Keiser Rig has reached 1500 h. This initial set of exposures has validated the use of very high H_2O pressures (18 atm) to evaluate EBC volatility, even at the slow-flow gas velocities used in the Keiser Rig. A new set of exposures is now being conducted on a more controlled set of samples.

Technical Progress

Several proof-of-principle specimens were included in the initial 1500 h high water-vapor-pressure exposures to demonstrate and measure volatilization in the Keiser Rig. CVD SiC and quartz have been well-characterized after exposure to elevated water vapor-pressures and were included as standards, and, since the primary purpose of these experiments was to measure volatilization of currently-used, state-of-the-art EBCs, hot-pressed BSAS, BAS, SAS coupons were exposed simultaneously with the Si-based standards. The mass and dimensions of each were determined before and after each exposure period (365, 500, 1000, and 1500 h). The gravimetric data for bulk specimens of BSAS, BAS, SAS, CVD SiC, SiO_2, after exposures at 1250°C showed that all three aluminosilicates exhibited similar kinetic behavior and had significantly lower total mass losses and rates of volatilization than the SiO_2 and CVD SiC. At times greater than 500 h, the volatilization rate of SiO_2 was approximately an order of magnitude greater than that for the mixed oxides. This difference is close to that expected based on results from combustor liner exposures. Quantitative estimates of recession rates under conditions where there is a significant flux of volatile species will require more detailed tracking of specimen dimensions and accompanying microstructural analyses, as will a mechanistic understanding of the volatilization process in multiphase ceramics structures. Nevertheless, the volatilization trends are clearly differentiated using the gravimetric data. In support of the theoretical treatment, summarized in previous Quarterly reports, these results provide an experimental proof-of-principle of using very high-pressure, low-gas-velocity exposures for qualitative differentiation of volatilization resistance among different candidate materials being developed for EBC applications. An added benefit of doing such at high H_2O pressures (versus low pressures, but high gas velocities) is that permeation resistance can also be evaluated (using better-than-worst-case conditions because the p_{H2O} is so high) when coatings on Si-based ceramic substrates are exposed.

Status of Milestones

Report the volatilization results from the exposure of 3 different Si_3N_4 compositions to very high H_2O-pressures in ORNL's Keiser Rig. June 2005 – on track

Industry Interactions

1. November 15-16, 2004. Charles Lewinsohn (Ceramatec) visited ORNL to discuss collaboration between ORNL/Ceramatec/St. Gobain to evaluate stability of Ceramatec EBCs in Keiser Rig.
2. November 17-18, 2005. Attended EBC Workshop in Nashville, TN. Presented "Evaluation of EBCs in ORNL Keiser Rig." Met with several industrial/academic contacts at meeting to discuss collaborative EBC evaluation work (Univ. of CO, St. Gobain Ceramics, ATK-COI Ceramics, etc.)

Characterization of Oxidation Resistant Ceramics and Silicon Nitrides

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Objectives

1) Determine the tensile creep properties of Saint Gobain's latest NT154 grade silicon nitride to ensure that this batch of material has at least equivalent creep properties to the previous material made in the mid 1990s.
2) Characterize the oxidation behaviour of candidate ceramics and two phase silicon nitrides in an air/steam environment to eliminate the need for complex costly multilayer coatings. The goal of this activity is to find materials that can be used in the construction of long life microturbine hot section components that enable the engine to operate at high efficiencies with good economic payback.

Highlights

Oxidation of $Lu_2Si_2O_7$ and $Sc_2Si_2O_7$ in flowing air/ water vapor at 1500°C for 140 hours did not affect the $Sc_2Si_2O_7$, whereas the $Lu_2Si_2O_7$ showed a minor weight loss of 0.3%. The greater stability of $Sc_2Si_2O_7$ is due to the smaller ionic size of the Sc3+ ion.

Technical Progress

Samples of $Lu_2Si_2O_7$ and $Sc_2Si_2O_7$ were prepared by hot pressing at 1800°C, and their thermal expansion coefficients determined. Both silicates were stable in air at 1500 °C. In an air/water vapor atmosphere the $Sc_2Si_2O_7$ was stable, but the $Lu_2Si_2O_7$ lost 0.3% by weight after 140 hours.

Several O' sialons (based upon the silicon oxynitride structure) were prepared with varying aluminum contents by hot pressing at 1800°C. Oxidation studies in air and air/water atmospheres showed formation of thin mullite containing layers interspersed with bubbles. Oxidation curves thus show both weight gain and weight loss. This loss of nitrogen appears to increase with increasing amounts of aluminum in the solid solution probably associated with increased formation of a nitrogen containing glass.

Status of Milestones

Determine the oxidation behavior of two phase silicon nitride materials in air/ steam environment at 1500°C. September 2005 – on track.

Industry Interactions

Discussions with several Industry researchers and managers at the recent EBC workshop in Nashville, TN on November 17-18, 2004.

Ceramic Composite Materials Characterization

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Objective

SiC/SiC continuous-fiber ceramic composite (CFCC) combustor liners with a BSAS-based environmental barrier coating (EBC) have been exposed in several Solar Turbines engine tests for >10,000 h. The engine-exposed combustor liners have been characterized microstructurally and mechanically at ORNL to evaluate degradation of both CFCC liner materials and EBC system. Simulated exposures of similar materials systems have been conducted simultaneously in ORNL's Keiser Rigs at high water-vapor pressures. More recently, new EBC compositions and CFCC liner (including oxide/oxide and another SiC/SiC) have (or will be) exposed in the Solar Turbines engines as well as in the Keiser Rig and will be evaluated post-exposure. The primary objective of this project is to understand degradation mechanisms of the various EBC and CFCC materials in combustion environments (elevated H_2O pressure).

Highlights

Microstructural characterization of three different engine-exposed EBC/CFCC combustor liners has been completed and the results have been presented to Solar Tubines, UTRC, Goodrich Corp., GE Power Systems Composites, and Argonne National Laboratory. Several CFCC/EBC processing issues and new materials systems were being evaluated in these liners; two of the three liners had new SAS-based EBCs and one had a 2-layer BSAS-based EBC (as opposed to the typical 3-layer system); the two liners with SAS-based EBCs did not have the standard CVD SiC seal coat applied during CFCC liner processing (possible cost reduction).

Technical Progress

During this Quarter, the following engine-tested (all < 10,000 h) liners were evaluated:

1. Inner liner from Texaco #6 engine test after 5135 h – MI SiC/SiC (Hi-Nicalon fibers) liner with Si/BSAS EBC (~200 μm CVD SiC seal coat)
2. 2. Inner liner from Malden Mills #3 engine test after 8368 h - MI SiC/SiC (Tyranno fibers) liner with Si/SAS EBC (no CVD SiC seal coat)
3. 3. Outer liner from Malden Mills #3 engine test after 8368 h – MI SiC/SiC (Tyranno fibers) liner with Si/(mullite+SAS)/SAS EBC (no CVD SiC seal coat). In general, several consistent degradation mechanisms were observed.

The liners with SAS-based EBCs (both 2-layer and 3-layer) performed very poorly due to (1) poor initial processing of the SAS layers (excessive porosity and inhomogeneous coating thickness) which allowed for significant gas-path CFCC oxidation/recession and (2) excessive back (cool)-side CFCC oxidation as a result of no CVD SiC seal coat. The inner liner with the 2-layer Si/BSAS EBC performed poorly due to excessive reaction between the BSAS and silica (somewhat expected based on known eutectic reactions at liner operating temperatures). Mechanical characterization is also being conducted on the liners.

Status of Milestones

Prepare a report and present results on the expanded use of ORNL's Keiser Rig to evaluate the volatility resistance of EBCs. *August 2005 – on track.*

Industry Interactions

1. November 10, 2005. Meeting held at Solar Turbines, San Diego to review characterization results from three recent engine-exposed CFCC combustor liners with SAS-based EBCs.
2. Visit by Irene Spitsberg, GEAE, to discuss collaborative work on EBC characterization.
3. November 17-18, 2005. Attended EBC Workshop in Nashville, TN. Presented "Evaluation of EBCs in ORNL Keiser Rig." Met with several industrial/academic contacts at meeting.

DEVELOPMENT OF MONOLITHIC CERAMICS AND HIGH-TEMPERATURE COATINGS

Kennametal's Hot-Section Materials Development

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Objective

Objective Determine potential of an existing structural sialon that is being manufactured for other applications that, commensurate with the requirements of advanced microturbines shows potential for strength, environmental stability, and manufacturability for complex shapes.Brief objective of the project.

Ceramic hot section components will enable higher microturbine operating temperature increasing fuel to electric conversion efficiency of 40%.

Highlights

Sialons ab831 and ab531 selected for additional mechanical characterization exhibited strength and fatigure resistance required for microturbine hot section components.

Technical Progress

Sialon ab531 has 28% alpha sialon. The beta sialon 'z' value is approximately 0.9. The room temperature flexure strength is 912 MPa with a 13.4 weibull modulus. At 1204 C the strength is 644 MPa with a 17 weibull modulus. The second sialon ab831 has an alpha content of 32%. The beta sialon 'z' value in ab831 is 0.3. The lower Al and O substitution compared to ab531 results higher aspect beta grains and a greater amount of grain boundary phase because less rare earth enters the alpha structure. The room temperature strength of ab831 is 875 MPa with a weibull of 7. At 1204 C the strength is 588 MPa with a 7.8 weibull modulus. .

The dynamic fatigue exponents at 1204 C range from 178 to 40 demonstrating that sialons can meet the structural requirements of the microturbines in this program that have a target operating temperature of 1150 C to 1250 C.

Status of Milestones

1) Sialon compositional preliminary matrix. Completed
2) Complete mechanical and microstructural characterization of initial Sialon compositions. September 2004 - Complete
3) Colloidal Processing of Silicon Nitride. April 2004 - Complete
4) Role of Microstructure and Crystalline Phase Evolution on Thermo-mechanical Properties of Sialon Ceramics for Microturbine Applications. October 2004 - Complete

Industry Interactions

Attend the EBC Workshop Nashville TN, November 17-18, 2004

Visit UTRC February 23, 2005.

Hot-Section Materials Development for Advanced Microturbines

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Objective
The goal of this program is to develop and optimize a high temperature silicon nitride based ceramic material and process suitable for microturbine hot section component applications.

Highlights
As-processed (AP) and bulk flexure strengths of samples cut from NT154 Integrally Bladed Rotor (IBR) were measured at ORNL and found to be equivalent to those obtained from test tiles. Bi-axial flexure AP and bulk strengths were measured to be 706 MPa and 960MPa, respectively.

Technical Progress
Four radial design rotors have been manufactured utilizing the new HIP process for improved AP surfaces. Excellent average part-to-part variation of 0.003", concentricity of 0.004", and average surface roughness of 30μin were achieved. Biaxial flexure AP strengths of samples taken from the blade and hub region were measured laterally to be 596 and 706 MPa, respectively. Fully machined biaxial flexure discs cut from IBR shaft section were also tested with a strength and Weibull modulus of 960 MPa and 24.4, respectively. This series of tests suggest that the mechanical property data generated from test bars have been reproduced in net-shape components.

Internal EBC development has focused on identifying and hot pressing novel oxide-based EBC compositions. Initial evaluation of the monoliths includes the measurement of CTE and recession resistance using an internal rig. Some promising candidates have been identified for coating application and densification development. Collaborations with Ceramatec, UTRC and ORNL are on-going.

Status of Milestones
Develop a suitable surface modification procedure (HEEPS, PC) or EBC for test tiles and components to improve the recession resistance. December 2004 - on-going

Demonstrate improved slow crack growth properties of NT 154, which are consistent with gas turbine designer needs. July 2005

Industry Interactions
1) Vimal Pujari and Ara Vartabedian attended and presented at the EBC Workshop in Nashville, TN on November 17th and 18th.
2) Numerous conversations with Ceramatec regarding EBC development.
3) Vimal Pujari met with Rishi Raj to discuss EBC development activities.
4) Discussions with Dave Stinton and Terry Tiegs (ORNL) on the proposed Phase III program statement of work.
5) Discussions with Bill Tredway and John Holowczak of UTRC on silicon nitride microturbine rotors.

Environmental Protection Systems for Ceramics in Microturbines and Industrial Gas Turbine Applications - Part A: Conversion Coatings

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Objective

The use of advanced structural ceramics, such as silicon nitride (Si_3N_4), in gas turbine engines can allow operation at higher temperatures, thus improving engine efficiency. However, in the combustion engine environment, Si_3N_4 can undergo rapid degradation due to the corrosive and erosive effects of high temperature, high pressure, high gas flow rate, and the presence of water vapor. A protective environmental barrier coating (EBC) is needed for Si_3N_4 to allow these advanced materials to be utilized in microturbines and industrial gas turbine engines. The goal of this effort is to develop a method for producing a protective coating on Si_3N_4 and to evaluate the ability of the coating to protect the ceramic in an engine environment.

Highlights

A coating of $Yb_2Si_2O_7$ + SiO2 was formed by pack cementation on Saint-Gobain NT154 silicon nitride and provided protection to the substrate when compared to uncoated material in a preliminary exposure test in a high-temperature, high-humidity environment.

Technical Progress

Recent efforts have been successful in producing pack cementation coatings that contain Yb-based and Sr-based compounds that are candidate materials being evaluated for Si_3N_4 EBCs. The rare earth garnet $Yb_3Al_5O_{12}$ was formed on NT154 Si_3N_4 by coating in a powder pack of Yb_2O_3 + Al_2O_3 at 1400ºC for 2 hrs. in Ar gas. A coating of $Yb_2Si_2O_7$ + SiO2 was formed on NT154 by embedding the sample in a pack of Yb_2O_3 and heating to 1400ºC for 2 hrs. in air. This coating provided protection to the Si_3N_4 substrate in a preliminary exposure test. The Sr-based compound $SrAl_2Si_2O_8$ (SAS) was formed in coatings on SN282 and AS800 Si_3N_4. SAS is being evaluated for EBCs as an improvement over the barium-containing compound BSAS, which has been used successfully to protect SiC substrates. The SAS compound was formed by using a powder pack of $Sr(NO_3)_2$ + Al_2O_3 and processing at 1400ºC for 2 hrs. in Ar.

New NT154 Si_3N_4 material that has been received form Saint-Gobain and will be used to prepare coated specimens for environmental exposure testing in the Keiser rig.

Status of Milestones

Assess the uniformity of surface conversion coatings applied to complex shaped ceramic substrates and evaluate the protective capability in the Keiser Rig. September 2005 – on track

Industry Interactions

1. Met with Irene Spitsberg of GE Aircraft Engines at ORNL to discuss EBCs for SiC/SiC composites.
2. Had discussions with Vimal Pujari and Ara Vartabedian of Saint-Gobain at the EBC Workshop concerning pack cementation coating of NT154 Si_3N_4.
3. Received additional samples of NT154 Si_3N_4 from Saint-Gobain for evaluation in pack cementation coating experiments.

Environmental Protection Systems for Ceramics in Microturbines and Industrial Gas Turbine Applications: Slurry Coatings

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Objective

In order to be cost competitive, microturbines will have to meet aggressive durability targets. Ceramic components without an EBC will not be able to meet the goals of > 20,000 operation hours. An EBC may enable these components to meet the expected lifetimes provided the EBC can be applied at low cost. The goal of this project is to continue to develop a low cost, slurry-based process to apply protective coatings for silicon based ceramic materials for use in microturbine and/or industrial gas turbine applications. This effort will be coordinated with industrial partners to assist in the development of an ideal coating material or material system for steam and high velocity resistance.

Highlights

Slurry development of candidate material systems has begun. Initial coated samples have been submitted for simulated exposure and characterization.

Technical Progress

Candidate materials systems were identified. The materials were either synthesized, acquired from commercial sources, or are in the process of being acquired. Surface charge characterization was completed on materials received to date. Rheological characterization was initiated on materials received in order to refine slurry parameters for dipping experiments. Iterations of coating, sintering, and characterization to minimize sintering temperatures and maximizing coating uniformity, desired thicknesses and densities are in process. Bend bars of AS800, SN282, and NT-154 that were determined to be successfully coated were submitted for simulated exposure testing in HT Lin's steam rig.

Status of Milestones

Evaluate the corrosion resistance of rare earth doped silicate materials in a simulated combustion environment. September 2005 – on track

Industry Interactions

Attended the Third Annual EBC Workshop at the Gaylord Opryland Resort & Convention Center in Nashville, TN on November 17-18, 2004. The presentation, "Slurry Based Environmental Protection Systems" was given on Thursday, Nov 18, 2004 by postdoctoral candidate, GH Kirby. Communications with Dr. Charles Lewinson and Dr. Shekar Balagopal at Ceramatec to discuss utilization of ORNL's colloidal expertise.

Polymer Derived EBCs for Monolithic Silicon Nitride

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Objective

The need for environmental barrier coatings (zirconia/ hafnia) for silicon nitride turbine components exposed to hot humid active oxidation conditions is well established. Polymer derived silicon carboxynitride based composites are candidate materials for bond coats between silicon nitride and the protective top coats because they show good adhesion to these materials and the composite microstructure can be tailored to accommodate the thermal expansion mismatch stresses. The aim of the present study is to optimize (polymer derived) Silicon Carboxynitride (SiCNO) composite bond coat for silicon nitride from studies on particulate composites made from transition metal oxides (hafnia/zirconia) and SiCNO.

Highlights

Weight change studies during the hydrothermal oxidation of (polymer derived) SiCNO – Hafnia/Zircona composites indicated a weight stabilization at longer exposure times. Studies on silicon nitride as a function of vapor velocity and studies on SiCNO under passive oxidation conditions have revealed that this stable weight region could be related to the decreased volatilization due to reduced vapor velocity as the steam flows through the porous structure. These observations strengthen the idea that the porous composite exhibits self-healing structure.

Technical Progress

In order to understand the reason for the stabilization in weight of the SiCNO – 50 vol% Hafnia/Zirconia composites, passive oxidation studies were conducted on SiCNO powders as a function of vapor partical pressure. These data revealed that SiCNO shows a parabolic weight gain. Studies were also conducted on silicon nitride as a function of vapor velocity at ~ 100% vapor partial pressure. These data showed that the volatilization rate varied as square root of velocity at velocities > 4 cm/sec but was significantly lower at a velocity of 1 cm/sec. This raises the possibility of a thresh-hold velocity for volatilization and experiments are currently underway to test this hypothesis.

The reason for the weight stabilization can be related to the decrease in the vapor velocity as the steam percolates through the pores of the composite: This would cause the silica near the surface to volatilize, whereas, the silica formed in the interior would not; leading to weight stabilization.

Three point bending tests were conducted on the hydrothermally tested samples. Microstructural examination of the fracture surfaces revealed that failure was due to shrinkage caused by oxidation of the coarse SiCNO particles to form silica. Based on these observations, new composite microstructures have been designed and are currently being tested.

Status of Milestones

2. Design a robust self-healing microstructure from studies on porous SiCNO based composites, and test its behavior under active hydrothermal oxidation conditions. May, 2005; on track

Industry Interactions

1) Presented these results at the 29th International Conference on Advanced Ceramics and Composites, Jan 23-28, 2005. Cocoa Beach, FL. 2) Presented results at EBC-Nashville Meeting in Nov'04. 3) Presented results at Honeywell in Jan'05. 4) Discussed possible interaction with St. Gobain.

EBC Development for Silicon Nitride Ceramics for Enhanced Hydrothermal Corrosion Resistance

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Objective

The goal of the current program at Ceramatec is to develop a functional environmental barrier coating system for silicon nitride ceramics, processed using non-line of sight techniques, which will provide substantially improved hydrothermal corrosion resistance without degradation of bulk mechanical properties.

Effective protection of silicon nitride ceramics from hydrothermal corrosion will enable components for microturbine engines, manufactured from these materials, to be operated at conditions that will provide improved efficiency for microturbine power generation systems.

Highlights

Multilayer coatings were fabricated on silicon nitride coupons by a low-cost dip-coating.

Technical Progress

Low silica activity coating compositions, selected from available hydrothermal stability diagrams, were screened by short-term hydrothermal exposure testing at Saint-Gobain, Northboro R & D Center. Specimens of the top coat materials were also exposed to hydrothermal conditions at the Oak Ridge National Laboratory, but specimen porosity One of the promising compositions was selected for coating development work. Bond coat materials, derived from pyroloysed preceramic polymer materials were developed to provide adhesion to silicon nitride substrates and accommodation of property mismatches between substrates and top coat materials. Multilayer coatings, consisting of graded bond coats and the selected low silica activity material were successfully deposited on silicon nitride coupons and bend bar specimens. Flexural strength measurements of the coated bend bars were made at room temperature and the results demonstrated that the coatings and coating process did not degrade the strength of the materials.

Status of Milestones

Industry Interactions

Frequent conference calls and e-mail communications occur between Carematec, Inc. and Dr. Vimal Pujari and Mr. Ara Vartebedian at Saint-Gobain, Ceramics and Plastics.
Visit to Ceramatec, Inc. by D. Stinton, October 21, 2004.

Attended the EBC Workshop on November 17-18, 2004 and discussed polymer-derived EBC with the attendees.

Failure Mechanisms in Coatings

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Objective

Provide critical information on processing, mechanical/microstructural behavior, and resulting failure modes/mechanisms of new and emerging environmental barrier coatings (EBCs). This information is critically needed for EBC processing and design for improved component performance that will result in increased turbine efficiency.

Correlate processing parameters and microstructure with performance in service environment to optimize processing and design of EBCs with improved performance.

Highlights

Evaluation of thermal fatigue resistance of a set of BSAS EBC–coated Si_3N_4 specimens obtained from United Technologies Research Center was completed. These results will be used to optimize microstructure and composition of novel EBCs.

Technical Progress

Since EBC-coated components are expected to experience thermal cycles in service, an evaluation of thermal fatigue resistance of novel and developing EBCs has been initiated. This quarter, thermal fatigue testing was performed on BSAS EBC-coated SN282 Si_3N_4 substrates. The test consisted of thermal cycling the BSAS EBC-coated substrates and characterizing the damage in the EBC after a predetermined number of thermal cycles. Each thermal cycle consisted of heating the coated substrates to 1250°C and holding at temperature for 0.5 hour to attain thermal equilibrium, followed by forced air cooling. Damage evaluation was performed by measuring elastic modulus of the various EBC layers by indentation technique before and after thermal cycling. Initially, the elastic modulus increased with increasing thermal cycles due to sintering and densification of the EBC layers. Subsequently after ~75-100 cycles, the modulus started to decrease resulting from microcracking of the EBC. A microstructural evaluation of the thermally cycled EBCs indicated that damage evolution in EBCs during thermal fatigue results from initiation, growth, and coalescence of numerous microcracks, which causes eventual spallation.
A comparison of thermal fatigue resistance of various EBCs will provide guidance for optimizing processing and selecting appropriate EBC for specific applications.

Status of Milestones

1. Complete evaluation of thermal fatigue resistance of BSAS EBC-coated Si_3N_4 specimens in oxidizing environment. November 2004 – completed.
2. Complete evaluation of thermal fatigue resistance of Ta_2O_5 EBC-coated Si_3N_4 specimens in oxidizing environment. February 2005 – on track.

Industry Interactions

1. Communications with Dr. Balky Nair at Ceramatec, Inc. to discuss the nature of novel bond coat based EBCs developed at Ceramatec and specific test to be performed at Argonne to characterize these EBCs.

POWER ELECTRONICS

Graphite-based Thermal Management System Components for Microturbine Heat Recovery Systems

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Objective

Because of low efficiency in power generation, as much as 90 GWh of energy are lost in the United States every year. However, by recovering waste heat, the efficiency of power generation systems can be significantly improved. In the case of recuperated microturbines, heat in the exhaust gases can be captured to dry humid air and/or to produce hot or chilled water for use in space heating, or air conditioning. The objective of this project is to design, fabricate and evaluate graphite fiber-based heat recovery systems for microturbines, which currently use aluminum fin heat exchangers. Through modeling and experimental work, the architecture of woven fiber structures that maximize heat transfer while minimizing pressure drop and cost will be identified.

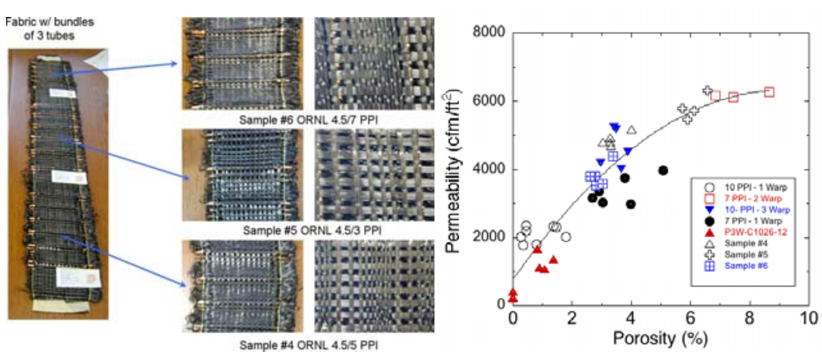
Highlights

Graphite fiber woven structures incorporating copper tubing were manufactured in collaboration with 3-Tex, Inc. Woven structures with different fiber architectures and permeability were obtained.

Technical Progress

Fabrics with varying pore structures were woven by 3-Tex, Inc. using graphite fibers with moderate modulus and thermal conductivity. Different weaving modes were utilized to produce continuous woven structures with either one or there layers of ¼”-diameter copper tubing integrated in the structure. The picture below depicts one of the structures obtained. In collaboration with the Textile and Nonwovens Development Center (TANDEC) at the University of Tennessee air permeability measurements were made on these structures. Permeability values ranged from 225 to 6250 cfm/ft^2.

A test set-up is being developed to evaluate, simultaneously, the permeability and transport characteristic of these structures.



Status of Milestones

Complete prototype fabrication of advanced heat exchanger using high conductivity fibers. June 2005 - on track

Industry Interactions

Worked with 3-Tex, Inc. (Rutherford, NC) and the Textile and Nonwoven Development Center (TANDEC) at the University of Tennessee-Knoxville.

Heat Exchange Concepts Utilizing Porous Carbon Foam

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Objective

Characterization of the fluid flow and heat transfer in porous carbon foam is required to enable engineers to utilize the material in heat transfer devices. Porous carbon foam has high specific and effective thermal conductivities and an open interconnected pore structure that enables heat to be rapidly transferred to infiltrated air. The current effort combines analysis, modeling and experiments to characterize the thermal and hydrodynamic performance of the foam for the purpose of developing correlations that can be used in the design of devices for engineering applications.

Highlights

Three technical papers were written this period. One technical paper published, one under review in *ASME Journal of Heat and Transfer*, one under review in *Journal of Applied Heat Transfer*.

Technical Progress

An analytical model has been developed to express the effective thermal conductivity, the permeability and the internal and external exposed area in terms of the porosity and pore diameter. The geometric model is based on a unit-cube geometry, which accurately represents the internal structure of the foam. The thermal model is developed by creating a thermal-electric analog in which the volumes of solid and fluid within the unit-cube are preserved. The model expressions are shown to give predictions within 10% of measured results

A small-scale wind tunnel facility was used to conduct experiments of parallel airflow across thin layers of foam bonded to an aluminum substrate. The foam layers were shown to enhance the convective heat transfer 10-40% depending upon the flow speed. The heat transfer enhancement was also shown to depend on the openness of the foam, i.e. higher enhancements were observed for more open foam structures.

A computational fluid dynamics model has been developed to compute the fluid flow and heat transfer in applications of conjugate solid/fluid/porous domains. The thermal model for the porous media is base on a non-equilibrium formulation of the energy equation whereby separate energy equations are solved for the solid and fluid components of the porous domain. Interface conditions that connect the solid/fluid/porous domains have been developed that are accurate for a wide range of flow Reynolds numbers and temperature differences.

Status of Milestones

1. Develop engineering model for new-generation air-water heat exchanger for application in heat recovery and aftercooling. Mar 2005 – On track.

Industry Interactions

1. Meetings were held at the Caterpillar Tech Center in Peoria to discuss modeling efforts and applications of porous carbon foam in heat dissipation devices.

MATERIALS FOR ADVANCED RECIPROCATING ENGINES

Characterization and Development of Spark Plug Materials and Components

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Objective

Spark plug lifetimes in advanced natural gas engines are on the order of only 1000-4000 h, which result in loss of performance and necessitate frequent, costly downtime and maintenance. Spark plug durability will become even more critical as future engines are pushed to leaner-burn conditions to reduce emissions. The goals of this effort are to gain insight into spark plug life-limiting wear processes, and to use this understanding to develop new electrode alloys to achieve lifetimes of ≥ 8000 h.

Highlights

A set of developmental electrode alloys were manufactured and delivered to Federal Mogul (FM) to be used to make spark plugs for engine testing at ORNL/NTRC.

Technical Progress

In collaboration with GTI, a series of spark plugs were run in a Cummins engine and removed after short-time intervals to gain insight into the onset of the degradation phenomena observed in end-of-life plugs. Preliminary analysis of 188h tested plugs showed oxidation/crack initiation at the Ni alloy electrode/Pt-W insert interface, and intergranular cracking in both of the Pt-W and Ir insert, similar to those observed in the end-of-life plugs. A series of engine-tested plugs from FM were also analyzed, and yielded similar findings.

Based on insights gained from the plug characterization, a set of model ferritic, austenitic, and Ni-base electrode alloys, which exhibit a range of CTE, electrical, and thermal conductivities, were designed to form an optimized Cr_2O_3 protective scale. These alloys were delivered to FM, and will be made into spark plugs for engine testing at ORNL/NTRC. Post-test characterization will be used to analyze the type and extent of degradation, to provide a basis to identify alloy characteristics that most influence attack of the electrode alloy/Pt alloy pad interface.

Status of Milestones

1. Characterize wear of currently used spark plugs as a function of time/ignition system, and engine exposure conditions to firmly establish key issues controlling wear in natural gas engines. Disseminate results in at least 1 open literature publication. Sept 2005 – on track
2. Manufacture electrodes from at least one new developmental alloy and evaluate and characterize under ignition conditions. Benchmark results compared to currently used Pt and Ir alloys. Sept. 2005 – on track.

Industry Interactions

1. Conference calls and communications with Drs. Iryna Levina and Jim Lykowski at FM to discuss the analysis results of FM spark plugs and update the alloy development status.
2. Communications with Dr. Gordon Gerber at Caterpillar to discuss the type of engine and plug specifications for spark plug evaluation task using ORNL developmental alloys.

Advanced Materials for Reciprocating Engine Components

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Objective

Next generation natural gas reciprocating engines will have higher in-cylinder pressures and temperatures in order to meet the goals of higher power density increased efficiency, and lower emissions. Component materials face the paradox of increased life and reliability while operating in higher temperatures that limit material performance and durability. In collaboration with ARES OEM's and their component suppliers, ORNL is characterizing the effects of long term engine exposure on intake and exhaust valves, their seats, and on exhaust components. With an appropriate baseline of current material/components mechanical and oxidation properties behavior, and underlying microstructural changes in various alloys, ORNL will work with the OEM's and their suppliers to establish the best materials and processing options for components with higher performance.

Approach

ORNL is focused on characterizing a series of intake and exhaust valves, and seats with long-term engine exposure to determine their performance limitations. This data will then be input to identify materials with more temperature capability and performance for such components. Similarly, ORNL is engaged with OEM's and foundries to compare exhaust components (manifolds, turbocharger casings) made from a new cast stainless steel, CF8C-Plus, with current cast-iron components.

Highlights

Characterization is nearly complete on an initial set of intake and exhaust valves with significant long-term ARES engine exposure.

Technical Progress

An effort began previously on Ni-based superalloy exhaust valve characterization, and expanded in 2004 include intake and exhaust valves and their seats. Comparison of fresh components with a series of components exposed to engine testing ranging from several thousand to up to twenty thousand hours continued this quarter at ORNL. While steel intake valves show little change so far, Ni-based superalloy exhaust valves show significant effects of aging after only a few thousand hours of engine service, including significant grain boundary $M_{23}C_6$ carbide precipitation and coarsening of the γ' precipitates that strengthen the alloy at high temperatures. The progression of such changes after longer engine exposure times will be shown next quarter.

This quarter, new discussions initiated between ARES OEM's and their exhaust manifold supplier to define a critical experiment to test the performance of the CF8C-Plus cast stainless steel relative to standard manifolds made of Ni-resist austenitic cast iron. If successful, this effort will produce exhaust manifolds to test side-by-side on an ARES test engine to determine performance benefits.

Status of Milestones

1. Complete initial characterization of fresh and engine-tested intake and exhaust valves, and their corresponding seats, to determine aging effects and degradation/failure mechanisms. January 2005 – on track

Industry Interactions

1. Conference calls and communications with principal investigators at OEMs or component suppliers occurs regularly (1-2 times/month) to guide this project.

Optimization of In-Cylinder Materials for Reciprocating Natural Gas Engines

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Objective

It is critical to define the operating environment of next generation reciprocating engines in order to identify required materials properties, assess new materials and/or materials processing to meet these engine-operating requirements, and to characterize the compatibility of materials with common natural gas lubricants in the new and used condition by use of advanced microstructural characterization techniques. In collaboration with Waukesha, a two-step approach is being taken to address the issue of oil deposit formation and loss of valve performance in ARES engines:

Objective 1

1.) Identify new materials or surface treatments that better resist oxidation and deposit formation leading to material loss. These new alloys should also have sufficient microstructural stability for extended high temperature exposure.

Objective 2

2.) Develop a strategy to prevent or minimize inorganic oil deposit formation.

Highlights

• Surface characterization was initiated on two sets of engine-exposed intake valves exhibiting significant deposit formation for comparison with intake valves characterized after ~2000 h engine use.

Progress

Objective 1

Techniques for preparing (sectioning/polishing) and quantifying the extent of metal oxidation and recession of engine-exposed intake valves with new (unused) intake valves have been investigated during the first quarter of FY 2005. Several intake valves, which were removed from engines run at varying times, have tulip and/or sealing surfaces exhibiting localized degraded surfaces (torched/burned), for an intake valve that ran in an engine for ~24,000 h. Two sections were cut from this particular valve through the valve sealing face and tulip region and were used to compare surface degradation from a "torched" area to a non-torched area (Figure 1). These cross-sections were also compared with the cross-sectional dimensions for a new intake valve. Surface recession (or loss) of the valve sealing face was determined by overlaying the outlines of the different engine-exposed valve cross-sections on top of the cross-section of the unused valve (Figure 2). Surface deposits were easily identified. Within the torched area, significant metal oxidation and metal recession was observed on the valve sealing face, creating a relatively wide gap where a valve/seat seal should be. As combustion gases leak into this gap, accelerated metal oxidation occurs with time. The initial cause of the torching appears to be due to catastrophic spallation of the (Ca,Zn) phosphate surface deposit adjacent to the valve/seat seal. In areas where torching is not evident on the valve sealing surface and/or tulip, no metal recession (limited metal oxidation) was observed. Additional intake valves are being characterized in this way to fully evaluate the "torching/burning" phenomenon on intake valve surfaces during engine use.

Objective 2

An effort was initiated in FY2004 to track oil degradation during engine operation and relate the degradation to the additive package of the oil and the efficiency of contaminant removal during engine operation through more advanced filtration. Comparison engine tests are currently being defined which will determine the relative contributions of oil composition and contaminant control to oil deposit formation which will enlist the efforts of an engine OEM, a major heavy duty filter manufacturer, and ORNL. An advanced oil test is currently being developed to measure the content of a heavy organic contaminant (sludge), which is thought to act as a precursor to oil deposit formation.

Thermal stability tests will be used this FY to determine the tendency of oils to form deposits as a function of composition and aging. In order to better develop lubricating oils for natural gas-fired engines that provide effective lubrication for extended service intervals, prevent or minimize the formation of in-cylinder oil deposits, and avoid the poisoning of exhaust catalysts, the formation of a consortium of the OEMs, lubricant and additive suppliers, and ORNL will be explored.

Milestones

1. Report on the characterization of current valve materials deposit formation as well as used oil. June 2005 – on track

Meetings

1. Conference calls and communications with Roger Rangarajan and Joseph Derra of Waukesha Engine Division to discuss the results of oil deposit characterization.

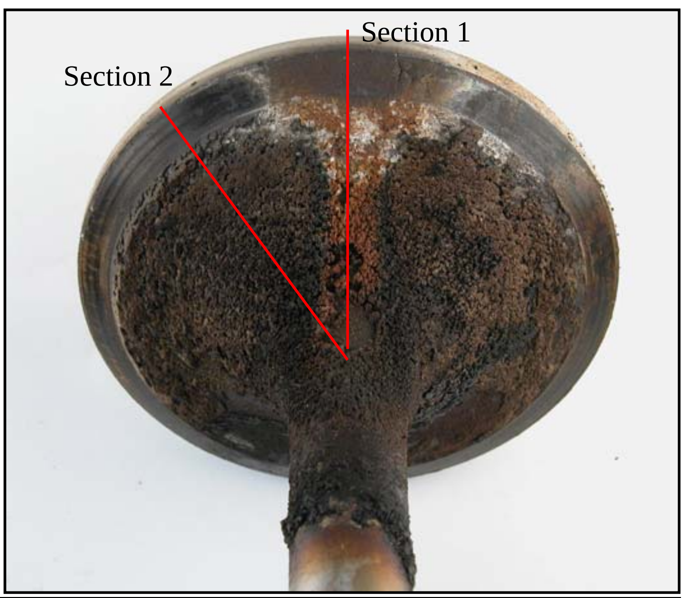

Figure 1. Intake valve removed from engine run for ~24,000 h showing sections cut for microanalysis.

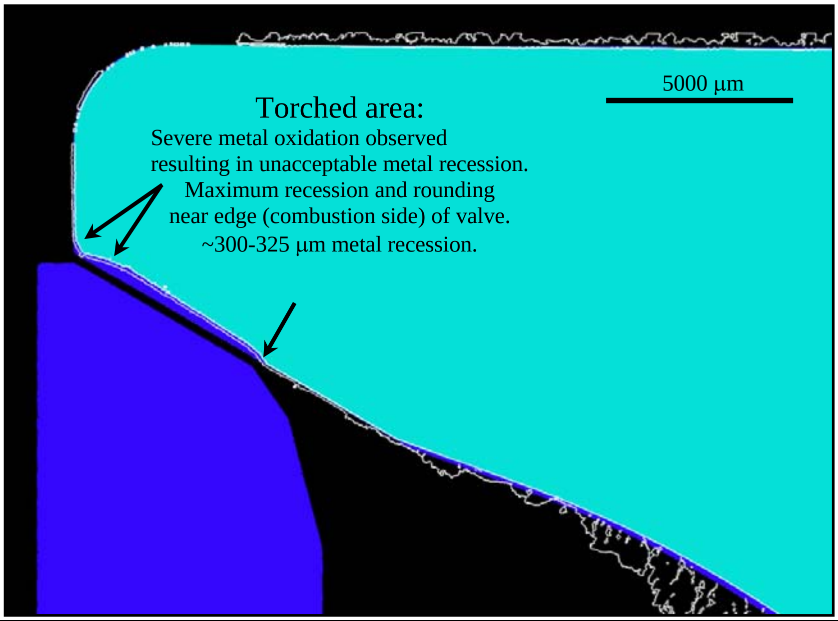

Figure 2. Section 1 from "torched" area of intake valve run in engine for ~24,000 h. Arrows designate valve sealing surface and area exhibiting metal recession.