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14. ABSTRACT This report summarizes the activities that were conducted as part of the AFOSR Workshop on Aerospace Materials for Extreme Environments that was held August 3-5, 2009 in Clayton (St. Louis), MO. The workshop brought together -80 participants from universities, government laboratories, and companies from around the world. The agenda included 28 invited presentations and 23 additional posters on the current status and latest research results related to the fundamental science of materials for possible application in hypersonic aerospace vehicles. This report includes a summary of workshop activities, a list of attendees, a copy of the foreword for the special issue of the Journal of the European Ceramic Society, and a list of presentation and poster titles. In addition, a CD will be submitted to the program manager with copies of presentations from the workshop.					
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Table of Contents

Table of Contents	1
Executive Summary	2
Objectives	3
Status of Effort.....	3
Accomplishments/New Findings	3
Personnel Supported	3
Publications/Presentations	4
Interactions/Transitions.....	4
Discoveries, Inventions, and Patent Disclosures	5
Awards	5
Appendix A: Attendance List	6
Appendix B: Workshop Schedule.....	8
Appendix C: Foreword for Journal of the European Ceramic Society.....	10
Appendix D: List of Manuscripts Submitted for Publication	11

Executive Summary

The Workshop on Aerospace Materials for Extreme Environments was held on August 3-5, 2009 in Clayton (St. Louis), MO. The workshop was attended by nearly 80 researchers from universities, government laboratories, and companies from around the world who investigate fundamental aspects of materials that may someday find use in advanced aerospace vehicles. The workshop consisted of ~25 invited presentations and ~20 additional poster presentations from leading researchers in this area from around the world. In addition to the current research leaders in this field, ~30 early-career professionals participated in the meeting in an effort to help attract a future generation of researchers to this challenging research field. Manuscripts based on workshop presentations will be published in an issue of the Journal of the European Ceramic Society focused on materials for extreme environments. Publication is planned in the late spring or early summer of 2010.

Objectives

The objective of this project was to organize and conduct a workshop focused on fundamental research related to ultra-high temperature structural materials that are candidates for use in hypersonic aerospace vehicles. The goal was to have participation from the leading researchers in the world who investigate ultra-high temperature materials for aerospace applications.

In addition to the primary goal of organizing the workshop, a secondary goal of the workshop was to help broaden participation in research related to ultra-high temperature materials for aerospace applications.

Status of Effort

The term of this project was from July 15, 2009 to November 30, 2009. As part of the project, The Workshop on Aerospace Materials for Extreme Environments was held on August 3-5, 2009 in St. Louis, MO.

The workshop was successful in attracting the leading researchers from the U.S. and the world who focus on ultra-high temperature materials for aerospace applications. The workshop was attended by nearly 80 researchers from universities, government research laboratories, and companies from the U.S., China, Italy, France, England, and the Ukraine.

The workshop was also successful in attracting participation from graduate students and early-career professionals who are the potential "next generation" of leaders in this field. The workshop was attended by ~30 early-career professionals and graduate students in an effort to foster their continued involvement with the community of researchers who investigate ultra-high temperature materials. A complete listing of attendees and their affiliations is provided in Appendix A.

Accomplishments/New Findings

The workshop included sessions on "Mechanical and Physical Properties," "Synthesis, Processing, and Structure," and "Oxidation and Environmental Response." A full workshop schedule is provided in Appendix B. The advancements in fundamental understanding of UHTCs related to these topics will be published as part of a focused section in an upcoming issue of the Journal of the European Ceramic Society.

Personnel Supported

Funds from the workshop were used to support the salary of a senior secretary in the Materials Science and Engineering Department at Missouri S&T (Table 1).

Table 1. Personnel supported on AFOSR contract FA2386-09-1-0564.

Name	Title/Role	Level of Support
Amy Moore	Senior Secretary	20 hours

In addition to salary for the senior secretary, funds from the workshop were used to provide travel support for invited speakers from foreign countries. Travel support was provided for several graduate students and select early-career professionals who participated in the workshop (Tables 2 and 3). The Asian Office of Aerospace Research and Development (AOARD, primary contact Dr. Kumar Jata) also provided funding that was used to partially support travel for the Chinese scientists.

Table 2. Invited foreign speakers who received travel support from AFOSR contract FA2386-09-1-0564.

Name	Title of Presentation/Other Contribution	Country
Laura Silvestroni	Toughened ZrB ₂ - Based Ceramics with Addition of SiC Whiskers or Chopped Fibres	Italy
Velerii Kotenko	New Methods of Strength Determination for Brittle Materials	Ukraine
Yanchun Zhou	New Zr(Hf)-Al(Si)-C Ceramics and Their Composites for Ultrahigh Temperature Applications	China
Bill Lee	Laser Modified Microstructures in ZrB ₂ , ZrB ₂ -SiC, and ZrC	England
Oleg Grygoriev	High-Temperature Ceramic Matrix Composites of MeB ₂ -MeSi ₂ Systems-Structure and Some Properties	Ukraine
Alina Ievdokymova	ZrB ₁₂ -Based Tool Materials for Titanium Alloys Machining	Ukraine
Volodymyr Filipov	Influence of Lattice Parameter Mismatch between Fibers and Matrix on Structure and Properties of Directionally Solidified LaB ₆ - (Ti,Zr)B ₂ Composites	Ukraine
Frederic Monteverde	Plasma Wind Tunnel Testing and Numerical Simulation of Ultra-High Temperature Ceramics for Space Applications	Italy
Xinghong Zhang	Preparation, Toughening/Strengthening and Oxidation of ZrB ₂ -Based Ultra-High Temperature Ceramic Composites	China
Guo-Jun Zhang	Microstructure Tailoring and Material Property Improvement of UHTCs	China

* Partial support for this participant provided through AOARD.

Publications/Presentations

Manuscripts based on presentations at the workshop will be published in a focused section of an upcoming issue of the Journal of the European Ceramic Society that is scheduled for publication in the late spring or early summer of 2010. The foreword drafted for the special issue is included as Appendix C while a list of manuscripts that have been submitted for publication is included as Appendix D.

Interactions/Transitions

The workshop provided a forum for informal interactions among researchers from academia, government research laboratories, and companies from the U.S. and abroad. The workshop provided a formal forum for researchers to present results to other leading researchers in this field, representatives from U.S. Department of Defense research laboratories, and companies that may utilize these materials in future aerospace vehicles. In addition, the participants had ample time for informal interactions during coffee breaks, lunches, and the afternoon reception in the Prologue Room at Boeing-St. Louis.

Table 3. Graduate students and early-career professionals who received travel support from AFOSR contract FA2386-09-1-0564.

Name	Institution	Poster Title
Xiao Ye*	Univ. of Mass.	Non-Contact Measurement of Creep Deformation
Luning Zhang†	SRI International	Thermal and Electrical Property Characterization of Diboride-Based UHTC Materials: Effects of Microstructure and Composition
Kalvis Terauds*	Univ. of Colorado	Piezoresistivity of Polymer Derived Ceramics at Elevated Temperatures
Jeremy Watts*	Missouri S&T	Stress Measurement in ZrB ₂ /SiC Composites using Raman Spectroscopy and Neutron Diffraction
Manuel Acosta*	Purdue	Design and Manufacture of Ultra-High Temperature Ceramics with Oriented Strengthening and Toughening Phases (CERASGEL)
Harlan Brown-Shaklee*	Missouri S&T	Reaction Sintering and Characterization of ZrB ₂ -SiC Composites
Matt Thompson*	Missouri S&T	Densification and Microstructure of ZrB ₂
David Lipke*	Georgia Tech	Reaction Syntheses of Discontinuous Tungsten Carbide Fibers
Luke Walker*	Univ. of Arizona	
Fei Ping† & Gregg Van Laningham	Georgia Tech	Oxidation Resistance of ZrB ₂ -Based Multi-phase Microstructures at Temperatures above 1500°C
Gaurav Mohanty* & Prasanna Balachandran*	Iowa State University	Informatics for Nanomechanical Characterization and Chemical Modeling of High Temperature Materials
Kathy Sevensen†	Valparaiso Univ.	A Comparison of the Oxidation Behavior of Hot Pressed and Spark Plasma Sintered HfB ₂ -SiC
P. Sarin†	Univ. of Illinois	In-situ Studies of Oxidation of ZrB ₂ and ZrB ₂ -SiC Composites at High Temperatures

* Graduate Student

† Early career professional

Discoveries, Inventions, and Patent Disclosures

None directly associated with this project.

Awards

None directly associated with this project.

Appendix A: Attendance List

	First Name	Last Name	Affiliation	Email Address
1	Manuel	Acosta	Purdue University	acosta@purdue.edu
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List continued on next page

Attendance List (Continued)

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Appendix B: Workshop Schedule

Monday August 3, 2009

Mechanical and Physical Properties

Time	Speaker	Title
1:00 pm	Joan Fuller	Welcome and workshop overview
1:15 pm	Laura Silvestroni	Toughened ZrB_2 -Based Ceramics with Addition of SiC Whiskers or Chopped Fibres
1:45 pm	Greg Hilmas	Modeling and Measuring Residual Stresses in ZrB_2 -SiC Ceramics
2:15 pm	Greg Morscher	Creep of SiC-SiC Composites
2:45 pm		Break
3:00 pm	Robert Hyers	Non-Contact Measurement of Creep in Ultra High Temperature Ceramics
3:30 pm	John Halloran	Electromagnetic Mechanical Apparatus for Non-Contact Mechanical Testing of Ultra High Temperature Ceramics
4:00 pm	Trudy Kriven	In situ Studies of High Temperature Phase Transformations in Oxide Ceramics
4:30 pm	Velerii Kotenko	New Methods of Strength Determination for Brittle Materials
5:00 pm		Poster Session
6:30 pm		Adjourn for the day

Tuesday August 4, 2009

Processing, Synthesis, and Structure

Time	Speaker	Title
8:00 am	Yanchun Zhou	New Zr(Hf)-Al(Si)-C Ceramics and Their Composites for Ultrahigh Temperature Applications
8:30 am	Bill Lee	Laser Modified Microstructures in ZrB_2 , ZrB_2 -SiC, and ZrC
9:00 am	Oleg Grygoriev	High-Temperature Ceramic Matrix Composites of MeB_2 - $MeSi_2$ Systems-Structure and Some Properties
9:30 am	Ken Sandhage	Near Net Shape/Dimension Fabrication of Refractory, Erosion-Resistant Carbide/Refractory Metal Composites
10:00 am		Break
10:30 am	Rishi Raj	Ultrahigh Temperature Semiconductors Made from Polymer Derived Ceramics
11:00 am	Dusan Pejakovic	Carbon-Rich Hafnia Thin Films Synthesized by Pulsed Laser Deposition
11:30 -1:00		Lunch Break on your own
1:00 pm	Alina Ievdokymova	ZrB_{12} -Based Tool Materials for Titanium Alloys Machining
1:30 pm	Donna Ballard and Don Weaver	Processing of Thin Gage Platinum Group Metal- Modified Nickel-Based Super Alloys and Gamma-TiAl
2:00 pm	Volodymyr Filipov	Influence of Lattice Parameter Mismatch between Fibers and Matrix on Structure and Properties of Directionally Solidified LaB_6 - (Ti,Zr) B_2 Composites
2:30 pm		Break

Tuesday August 4, 2009 (continued)

Processing, Synthesis, and Structure (continued)

3:00 pm	Art Bronson and Jack Chessa	Computational Design of Ultrahigh Temperature Ceramics
3:30 pm	Valeriy Kartuzov	Pseudopotential Method of Calculation of Temperature of Formation and Eutectic Concentration in System $\text{LaB}_6\text{-MeB}_2$ (Me -Ti, Zr, Hf, Cr, V)
4:00 pm	Inna Talmy	Synthesis, Processing, and Properties of TaC-TaB ₂ -C Ceramics
4:30 pm	Bridget Rogers	Formation and Characterization of AlO_xC_y and HfO_xC_y
5:00 pm	Igor Kartuzov	Estimation of Pulling Rate Interval in the Method of Directed Solidification of Fibrous Boride Composites
5:30 pm		Adjourn for the day

Wednesday, August 5, 2009

Oxidation and Environmental Response

Time	Speaker	Title
8:00 am	Frederic Monteverde	Plasma Wind Tunnel Testing and Numerical Simulation of Ultra-High Temperature Ceramics for Space Applications
8:30 am	Doug Fletcher	Status of the 30 kW Inductively Coupled Plasma Torch Facility at University of Vermont
9:00 am	Joe Marschall	Optical Diagnostics for UHTC Materials Characterization
9:30 am	Sylvia Johnson and Matt Gasch	Arcjet Oxidation and Mechanical Evaluation of Hafnium-Based Ultra-High Temperature Ceramics Fabricated by Hot Pressing and Field Assisted Sintering
10:00 am		Break
10:30 am	Robert Speyer	Oxidation Resistance of ZrB_2 with SiC, TaB ₂ , and TaSi ₂ Additions up to 1800°C
11:00 am	Erica Corral	Processing and High Temperature Testing of $\text{ZrB}_2\text{-C-C}$ Composites Coated with SiC
11:30 am	Xinghong Zhang	Preparation, Toughening/Strengthening and Oxidation of ZrB_2 -Based Ultra-High Temperature Ceramic Composites
noon-1:45		Lunch on your own
1:45 pm		Transportation from hotel to Boeing (meet in lobby)
2:00 pm		Boeing Prologue Room reception/ Large Core Arc Tunnel tour
4:30 pm		Transportation back to Hotel
4:45 pm		Arrive at hotel and adjourn

Appendix C: Foreword for Journal of the European Ceramic Society

Guest Editorial

Joan Fuller, Greg Hilmas, William Fahrenholtz, Erica Corral, and Laura Riegel

The manuscripts in this issue are based on presentations at the Workshop on Aerospace Materials for Extreme Environments that was held August 3-5, 2009 in St. Louis, MO. This was the third in a series of workshops sponsored by the United States Air Force Office of Scientific Research.¹ The purpose of the workshop was to discuss the current status and latest technical progress of fundamental research on ultra-high temperature materials. The workshop featured invited speakers from academia, industry, and government laboratories from around the world. The agenda included 28 presentations and 23 posters that were focused on the basic science of processing, microstructure development, properties, and modeling of ultra-high temperature materials.

Ultra-high temperature materials have been categorized based on wide-ranging criteria such as melting temperature ($>3000^{\circ}\text{C}$), upper temperature limit for continuous use ($>1600^{\circ}\text{C}$), or chemical family (refractory transition metals, carbon, plus carbides, borides, and nitrides of early transition metals). To be included in this workshop, materials had to be potential candidates for transition into future hypersonic air or space vehicles. Some of the likely applications for these ultra-high temperature materials include leading edges, acreage thermal protection systems, scramjet flow-path components, and rocket propulsion components.

Historically, ultra-high temperature materials were investigated in the U.S.S.R. and the U.S. from the late 1950s through the early 1970s as part of the original space race. Fundamental research on ultra-high temperature materials was largely dormant until a recent resurgence that began in the late 1990s. Since that time, research groups have been established in Italy, China, Ukraine, the U.S., and other countries. As an indication of the increase in the level of research worldwide, data available on Scopus indicates that publications on the topic of "ultra-high temperature ceramics" increased from ~5 in 2000 to 74 in 2008 while published papers on the compound zirconium diboride increased from ~80 in 2000 to more than 200 in 2008.

The Workshop on Aerospace Materials for Extreme Environments included sessions on "Mechanical and Physical Properties," "Processing, Synthesis and Structure," and "Oxidation and Environmental Response." Some of the significant accomplishments that are reported in the manuscripts in this issue include in-situ measurement of mechanical properties at temperatures above 2000°C , a crystal chemical approach to synthesis of ultra-refractory ternary carbides, and diagnostics for material behavior in plasma environments similar to those encountered in hypersonic flight and/or atmospheric re-entry. Taken together, the manuscripts and presentations point to the tremendous challenge associated with modeling, predicting, and controlling the behavior of structures with complex, hierarchical, and/or engineered architectures that are composed of materials with dissimilar physical and chemical properties.

Despite the significant progress reported in the manuscripts in this issue, discussions at the workshop pointed to several outstanding areas where additional research efforts are needed. In particular, research is needed to understand coupled thermal and mechanical behavior including the development of methods to monitor and predict highly correlated properties in extreme environments. Novel compositions and chemistries are also relatively underreported and new compositions with microstructures tailored for improved elevated temperature strength and creep resistance in addition to improved resistance to oxidation would greatly enhance future capabilities. Hopefully, future workshops will report on progress made in these and other areas.

¹. Manuscripts from the other two workshops were published in the Journal of Materials Science, 39(19), October 2004 and the Journal of the American Ceramic Society, 91(5), May 2008.

Appendix D: List of Manuscripts Submitted for Publication

Contact	Author	Authors and Title
Donna	Ballard	D.L. Ballard, D.S. Weaver, A.L. Pilchak and S.L. Semiatin, "Hot Working of Platinum Group Metal-Modified Ni-Base Superalloys"
Arturo	Bronson	H. Petla, E. Renova, A. Bronson, J. Chessa and N. Maheshwaraiah, "A Computational Analysis of a ZrO_2 - SiO_2 Scale for a ZrB_2 -ZrC-Zr System as an Ultrahigh Temperature Ceramic Composite"
Erica	Corral	E.L. Corral and L.S. Walker, "Inhibition of C-C Composites Using Zirconium Diboride and Boron Carbide"
Matt	Gasch	M. Gasch and S. Johnson, "Physical Characterization and Arcjet Oxidation of Hafnium-Based Ultra High Temperature Ceramics Fabricated by Hot Pressing and Field Assisted Sintering"
Oleg	Grygoriev	O. Grigoriev, B. Galanov, V. Lavrenko, A. Panasyuk, V. Kotenko, S. Ivanov, A. Koroteev, and K. Nikel, "Sintering and Microstructure of ZrB_2 -SiC($ZrSi_2$) System"
John	Halloran	S. Gangireddy, J. Halloran, and Z. Wing, "Non-Contact Mechanical Properties of UHTC at Ultrahigh Temperatures Using Lorentz Forces on Electrically Heated Ribbons"
John	Halloran	S. Gangireddy, S. Karlsdottir, and J. Halloran, "In Situ Optical Microscopy During High Temperature Oxidation of Zirconium Diboride- Silicon Carbide Ultra High Temperature Ceramic Composites, Part 1: Liquid Flow and the Growth of Zirconia Deposits"
John	Halloran	S. Gangireddy, J. Tucker, S. Norton, and J. Halloran, "In Situ Optical Microscopy During High Temperature Oxidation of Zirconium Diboride- Silicon Carbide Ultra High Temperature Ceramic Composites: Part 2- Formation of Carbon Monoxide Bubbles"
Greg	Hilmas	J. Watts, G. Hilmas, W. Fahrenholtz, D. Brown, and B. Clausen, "Stress Measurements in ZrB_2 -SiC Composites Using Raman Spectroscopy and Neutron Diffraction"
Greg	Hilmas	M.C. Teague, G.E. Hilmas, W.G. Fahrenholtz, "Mechanical Properties of Reactively Processed W/ Ta_2C -Based Composites"
Robert	Hyers	X. Ye and R.W. Hyers, "Computational Simulation of Non-contact Creep Deformation of ZrB_2/ZrB_2+SiC "
Sergey	Ivanov	O.N. Grigoriev, B.A. Galanov, V.A. Lavrenko, A.D. Panasyuk, S.M. Ivanov, A.V. Koroteev, and K.G. Nikel, "High-Temperature Oxidation of ZrB_2 -SiC-ZrSi ₂ Ceramics in Oxygen"
Valery	Kotenko	V. Kotenko, O. Grygoriev, B. Galanov, and S. Ivanov, "New Methods for the Strength Properties Determination of Brittle Materials"
Trudy	Kriven	P. Sarin, P. Driemeyer, R. P. Haggerty, D.-K. Kim, J. L. Bell, and W. M. Kriven, "In-Situ Studies of Oxidation of ZrB_2 and ZrB_2 -SiC Composites at High Temperatures"
William	Lee	D.D. Jayaseelan, H. Jackson, E. Eakins, P. Brown and W.E. Lee, "Laser Modified Microstructures in ZrB_2 , ZrB_2/SiC and ZrC"
Jochen	Marschall	T.H. Squire and J. Marschall, "Material Property Requirements for Analysis and Design of UHTC Components in Hypersonic Applications"
Jochen	Marschall	J. Marschall and D.G. Fletcher, "High-Enthalpy Test Environments, Flow Modeling and In Situ Diagnostics for Characterizing Ultra High Temperature Ceramics"
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List continued on next page

List of submitted manuscripts (continued)

Contact	Author	Authors and Title
Dusan	Pejakovic	D. Pejakovic, J. Marschall, M. George, B. Rogers, W. Nieveen, and V. Pajcini, "Synthesis of Carbon-Rich Hafnia Thin Films by Pulsed Laser Deposition"
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