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## BOOK OF ABSTRACTS

# 10th International Conference on High Temperature Ceramic Matrix Composites – HT-CMC 10

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# Topic: 1

# Computational modeling, simulation and design of new materials and processes

Topic: 1 - Computational modeling, simulation and design of new materials and processes
Abstract no. 2279

# Macroscopic Numerical Modeling of CVI Process for Preforms with Non-Uniformly Scaled Pores

Presenting author: Dr KULIK Alexey
Affiliation: STR Group, Inc. – Soft-Impact, Ltd.
64 Bolshoi Sampsonievskii pr.,Build. “E”.,194044 Saint Petersburg (Russia)
Co-authors: KULIK Victor (Baltic State Technical University “VOENMEH”), RAMM Mark (STR Group Inc. – Soft-Impact Ltd.)

Chemical Vapor Infiltration (CVI) is one of the most promising methods for production of high-quality Ceramic Matrix Composites (CMC). Generally, the preform to be densified in the CVI process is a complex porous medium with several systems of pores. Preforms composed of bundles of fibers contain two easily distinguishable pore systems. One of them is associated with pores formed by fibers in a bundle; another is a system of pores between the bundles. These pore systems have significantly different scales. As a result, all structural and transport properties of the porous media within these systems – pore size, specific surface, permeability, and species diffusion coefficients – noticeably differ. This leads to the fact that parameters optimal to densify different pore systems are also different. In this work, a macroscopic reactor-scale numerical model is developed to simulate CVI process accounting for presence of several pore systems in the preform. The model describes heat transfer in the whole reactor and flow of gas mixture in the reaction chamber including porous medium of the preform. Matrix material deposition in the pores as a result of heterogeneous chemical reactions is considered together with evolution of porous medium structure due to the preform material densification. In the present work, the model was used to study CVI process for production of SiC matrix composites from methyltrichlorosilane (MTS) CH3SiCl3. Variation of the material density and its distribution over the preform bulk for different pore systems and effect of operating conditions on the preform evolution were analyzed.

Topic: 1 - Computational modeling, simulation and design of new materials and processes
Abstract no. 2370

# Computational aided materials discovery for advanced ceramics


Presenting author: Prof ZENG Qing-feng
Affiliation: Science and Technology on Thermostructural Composite Materials Laboratory, School of Materials Science and Engineering, Northwestern Polytechnical Uni
No. 127 Youyi West Rd.,710072 Xi'an (Shaanxi, China)

Co-authors: ZHANG Li-tong (Science and Technology on Thermostructural Composite Materials Laboratory School of Materials Science and Engineering Northwestern Polytechnical University), CHENG Lai-fei (Science and Technology on Thermostructural Composite Materials Laboratory School of Materials Science and Engineering Northwestern Polytechnical University)



Rapid material design, manufacturing and evaluation technologies are urgently needed in rapidly changing industrial applications. The application of computer in material design is becoming more and more important. Especially for high-risk aerospace applications, through computer modelling and simulation of the structure and properties of new materials, the scientific application of new materials can be realized. This report shows our recent research progress in the computer-aided materials discovery and characterization, including the computational discovery of ultra-high temperature carbides, borides and nitrides ceramics, the simulation of their intrinsic properties and high temperature properties.

Topic: 1 - Computational modeling, simulation and design of new materials and processes
Abstract no. 2008

# Image-based modeling of the mechanical behavior of CMCs


Presenting author: Dr COUEGNAT Guillaume
Affiliation: Univ. Bordeaux - LCTS
3 Allée La Boétie,33600 PESSAC (Aquitaine, France)

Co-authors: BéNéZECH Jean (U. Bordeaux - LCTS), MAZARS Vincent (Université de Bordeaux - LCTS), CATY Olivier (Université de Bordeaux - LCTS), VIGNOLES Gerard Louis (Université de Bordeaux - LCTS)



This presentation will introduce some image-based modelling approaches that have been developed for the understanding and design of thermostructural materials such as fiber-reinforced SiCf/SiC composites. These tools are based on 3D images, either produced by tomography or by image synthesis. Combinations of original image processing methods and numerical simulation algorithms make it possible to study in detail in these classes of composite materials the relationships between structure and mechanical behavior, including the non-linear part with progressive damage.

Topic: 1 - Computational modeling, simulation and design of new materials and processes
Abstract no. 2205

# Continuous Fiber Reinforced Composite Microstructure Quantification via Mechine Learning: Understanding Fiber Chirality

Presenting author: Dr PRZYBYLA Craig
Affiliation: Air Force Research Laboratory
AFRL/RXCCP,45431 Wright-Patterson AFB (Ohio, United States)

Co-authors: HUNT Reece (Southwestern Ohio Council for Higher Education), SIMMONS Jeff (AIr Force Research Laboratory), PIERCE Jennifer (University of Dayton Research Institute), CHAPMAN Michael (UTC Inc.), UCHIC Michael (Air Force Research Laboratory), PRZYBYLA Craig (Air Force Research Laboratory)

As observed in nature, complex heterogeneous microstructures in materials like nacre can exhibit extraordinary resistance to fracture. In a similar manner, brittle matrix composites with continuous fiber reinforcement express a complex hierarchical microstructure that promotes toughness. However, little has been done to quantify how local microstructure heterogeneity influence fracture behavior in brittle matrix continuous fiber reinforced composites. To this end, a novel field variable has been developed to quantify fiber chirality or the amount of twist exhibited by bundles of continuous fibers through a volume of composite material. It is hypothesized that fiber chirality will have an influence on fracture behavior in composite materials. The approach is validated on phantom data and then employed to quantify the chirality in several brittle matrix composites. Using this technique, a unique substructure can be visualized in the microstructure as groups of fibers appear to migrate in unison relative to each other through the material volume on scales much smaller than the individual fiber tows. Data from both undamaged and damage composites are considered. The implications of the sub-structuring of the filaments on the fracture toughness of these class of composites is considered.

Topic: 1 - Computational modeling, simulation and design of new materials and processes
Abstract no. 2269

# Automated Structural Recognition and Evaluation of CMCs using Deep Neural Networks.

Presenting author: Prof SHICHIJO Naohiro
Affiliation: Tokyo University of Technology
1404-1 Katakura,Hachioji,192-0982 Tokyo (Japan)

Co-authors: SHICHIJO Naohiro (Tokyo University of Technology), TAKESHIMA Yuriko (Tokyo University of Technology), MATSUOKA Johei (Tokyo University of Technology), KANO Toru (Tokyo University of Technology), KAGAWA Yutaka (Tokyo University of Technology)

In this paper we propose a machine learning approach of efficiently segmenting constituent phases of Ceramic Matrix Composites (CMCs) including fibers, matrix and pores from microstructure voxel data obtained with X-ray micro-focus CT using deep neural networks (DNN). The correct dataset will be manually fabricated by inspector using 3D microstructure visualizer and be used as an input for DNN classifier during the learning phase. By using this approach, the labor productivity during inspection of manufacturing damage will be significantly lowered compared to currently used procedural machine recognition approaches.

Topic: 1 - Computational modeling, simulation and design of new materials and processes
Abstract no. 2274

# C/C-SiC Materials Based on High Performance C Fibres with Tailored Fibre-Matrix Bonding

Presenting author: M HEIDENREICH Bernhard
Affiliation: DLR
Pfaffenwaldring 38-40,D-70569 Stuttgart (Germany)

Co-authors: JAIN Neraj (DLR Institute of Structures and Design), KOSIN Marvin (DLR Institute of Structures and Design), KOCH Dietmar (DLR Institute of Structures and Design), SCHNECK Tanja (German Institutes of Textile and Fiber Research Denkendorf (DITF), CLAUß Bernd (German Institutes of Textile and Fiber Research Denkendorf (DITF), HERMANUTZ Frank (German Institutes of Textile and Fiber Research Denkendorf (DITF), BUCHMEISER Michael R. (German Institutes of Textile and Fiber Research Denkendorf (DITF), BASTIAN Brück (University of Augsburg Institute of Physics), SCHULZ Michael (University of Augsburg Institute of Physics), MüLLER Wolfgang (University of Augsburg Institute of Physics), HORN Siegfried (University of Augsburg Institute of Physics)

The Liquid Silicon Infiltration (LSI) process is used to manufacture carbon fiber based C/C-SiC composites. A CFRP-preform (carbon fibre reinforced polymer) is manufactured by infiltrating C fibre rovings with a phenolic resin, wet filament winding and warm pressing. After curing, the polymer matrix is converted to carbon by a high temperature treatment. Due to matrix shrinkage, the resulting C/C preform is characterized by a high porosity. The pore morphology includes open porosity in form of segmentation microcracks (SMC), caused by fibre bundle segmentation, fragmenting the fibre rovings into discrete C/C bundles and forming a 3D-network of interconnected microchannels, as well as fine, closed pores in the C matrix. Depending on the fibre matrix interface (FMI) and the strength of fibre matrix bonding (FMB), also debonding between individual filaments and matrix, so called fibre matrix debonding (FMD), is observed in the C/C bundles, forming additional open porosity all over the composite. During siliconization, the molten silicon is infiltrating the microchannels and open pores and SiC matrix forms by a chemical reaction of Si and C. In order to obtain damage tolerant C/C-SiC materials, the C fibres have to be protected from Si contact and conversion to SiC. Therefore, FMI has to be tailored to form dense C/C bundles and to avoid debonding between filaments and matrix.
In the current work, the formation of SMC and FMD is studied in CFRP-preforms based on standard high tenacity as well as on intermediate and ultrahigh modulus C fibres and is simulated in a FE-model. The influence of fibre surface treatment on FMB and microstructure development as well as on the material processing and, finally, on the mechanical properties of the resulting C/C-SiC materials is presented.

Topic: 1 - Computational modeling, simulation and design of new materials and processes
Abstract no. 2287

# Predicting the effects of microstructure on matrix cracking evolution in fiber reinforced CMCs via Machine Learning


Presenting author: Dr PATEL Dipen
Affiliation: AFRL/UES
Hobson Way,45324 Fairborn (Ohio, United States)
Co-authors: PARTHSARATHY Triplicane (AFRL/UES), PRZYBYLA Craig (AFRL)



A reduce-order, data-driven, probabilistic predictive model to quantify damage initiation in CMCs at pertinent lengths scales using a machine learning tools is proposed and explored. A novel data driven framework is developed to incorporate microstructural features using a statistical representation and using them to predict matrix crack initiation. The approach is illustrated for the case of transverse cracking in CMCs. A variety of fiber distributions are quantified using n-point statistics, and they are correlated with damage initiation statistics using artificial intelligence (AI) tools. Results from a FEM analysis are used as the ground truth to calibrate and validate the model.

Topic: 1 - Computational modeling, simulation and design of new materials and processes
Abstract no. 1920

# Effects of Defects on Delamination Failure in Ceramic Matrix Composites

Presenting author: Dr KUMAR Rajesh
Affiliation: United Technologies Research Center
411 Silver Ln,MS 129-30,06108 East Hartford (CT, United States)

Co-authors:

Ceramic Matrix Composite (CMC) materials can be subjected to high interlaminar stresses due to complex geometrical features (e.g., curvature) and/or transverse and multiaxial loads. Such high interlaminar stresses can initiate and propagate delamination cracks, especially in laminated (unidirectional and two-dimensional woven) architectures. Furthermore, as-produced CMC materials can have process-induced flaws and defect structures that can affect their mechanical properties. While there have been some studies on the effects of defects on the overall elastic properties of CMCs, relatively little research has been conducted on their effects on delamination failure mode. The present paper will discuss the results of computational models of delamination failure where defect structures are explicitly represented. Effects of defects on interlaminar tension, interlaminar shear, and both mode I and mode II delamination propagation will be discussed.

Topic: 1 - Computational modeling, simulation and design of new materials and processes
Abstract no. 2066

# Modeling the Oxidation Embrittlement of SiC/BN/SiC Composites in Water Vapor Environments


Presenting author: Mme COLLIER Virginia
Affiliation: University of California, Santa Barbara
UCSB Materials Department,Engineering II (Bldg 503) Room 1355,93106 Santa Barbara (California, United States)
Co-authors: BEGLEY Matthew (University of California Santa Barbara), XU Wenbo (University of California Santa Barbara), ZOK Frank (University of California Santa Barbara), MCMEEKING Robert (University of California Santa Barbara)



Oxidation embrittlement of SiC matrix/boron nitride interphase/SiC fiber ceramic matrix composites (SiC/BN/SiC CMC) at intermediate temperatures is a critical yet poorly described aspect of CMC durability. While the mechanical behavior of CMCs in inert environments is well-studied, the understanding of interactions between local chemistries and mechanics is lacking and remains a hurdle to wide-spread CMC implementation. Therefore, a model has been developed to probe three major oxidation phenomena: (i) diffusion of oxidants along a matrix crack; (ii) reaction of the BN interphase and diffusion of the gaseous boron hydroxide reaction products; and (iii) growth of an oxide scale along the fiber and matrix crack surfaces. Scaling arguments identify key rate-limiting species, e.g. the oxide growth rate is dominated by water vapor concentration and production of the lightest boron hydroxide product, HBO2, is negligible in comparison to the heavier reaction products---H3B3O6 and H3B3O6---at intermediate temperatures (700-900 C). Consequently, the model only tracks the concentration distribution of the heaviest boron hydroxide species in high water vapor environments. The coupling between transport-oxidation and mechanics is explored by calculating fiber-bridging stresses and crack opening displacements as a function of time in the evolving oxidized state. The effect of BN recession is accounted for by adjusting the shear sliding length of classical fiber bridging models. The results highlight key sensitivities to temperature and environmental conditions and provide insight to the interactions of local chemistries and mechanics.

Topic: 1 - Computational modeling, simulation and design of new materials and processes
Abstract no. 1986

# Estimation of the damage evolution of an oxide/oxide composite structure subjected to fatigue stress:comparison between tests and simulations


Presenting author: Mme SALLY Orianne
Affiliation: Safran Ceramics, a technology platform of Safran Tech
29 avenue de la Division Leclerc,92322 Chatillon (France)

Co-authors: LAURIN Frédéric (Onera DMAS Université Paris-Saclay), JULIEN Cédric (Onera DMAS Université Paris-Saclay), DESMORAT Rodrigue (LMT Cachan Université Paris-Saclay), BOUILLON Florent (Safran Ceramics a technology platform of Safran Tech)



Oxide/Oxide CMCs are considered for engine components subjected to intermediate thermo-mechanical loadings, such as civil aircraft plugs. One of their most interesting properties resides in the absence of dependence of the behaviour on temperature, consequently the non-linear response of this material is imputed exclusively to damage mechanisms.
Damage control and life prediction of composite parts constitute major challenges for civil applications. It is necessary to develop efficient calculation strategies for the design of composite parts submitted to both static and fatigue loadings. This study aims thus to propose computational strategies to forecast fatigue strength and fatigue lifetime of any given composite structure submitted to real loadings (complex and long-term ones). This study is focused on oxide/oxide 2D woven-ply laminates.
The present approach consists in proposing a damage model capable of determining the service life and residual properties of the material under static and/or fatigue loading, even complex ones. The model uses a kinetic formalism that allows complex loads to be taken into account without having to convert them into equivalent cyclic loads. However, the calculation costs are too high to simulate the behaviour of the structure for a high number of cycles.
Therefore, in a second step, a calculation strategy was developed in order to reduce fatigue calculation costs on large structure, and to make this model useable in design offices. A non-linear cycle jump method has been considered, which relies closely on the model damage law. This method has been successfully compared to conventional cycle skipping methods and has been implemented in a structural commercial finite element code. It allows to quickly obtaining the properties of a structure subjected to oligocyclic or polycyclic loadings. The error generated using the proposed cycle skip method is checked during the calculation, with respect to the reference solution obtained by simulating all cycles.

Topic: 1 - Computational modeling, simulation and design of new materials and processes
Abstract no. 1977

# Surface Recession of a 3D C/C composite Under Turbulent Flow

Presenting author: M LAMBOLEY Xavier
Affiliation: Laboratoire des Composites ThermoStructuraux
3 allée de la Boétie,33600 Pessac (France)

Co-authors: VIGNOLES Gérard (Laboratoire des Composites ThemoStructuraux), MATHIAUD Julien (Laboratoire des Composites ThemoStructuraux), COUZI Jacques (CEA), DUBROCA Bruno (Laboratoire des Composites ThemoStructuraux), NGUYEN-BUI Thanh-Hà (Centre Lasers Intenses et Applications)

During an atmospheric re-entry, the high temperatures (> 2000 K)
at the surface of the heat shield will lead to its surface recession due to the ablation by oxidation, nitridation and sublimation of the carbone/carbone composite. The development of the turbulence around the object will then induce the acquisition of surface roughness patterns known as ”scallops”.
The stability of these morphological features is studied, considering a perturbation in the viscous turbulent boundary-layer equations coupled to the diffusion-convection of oxydant, the Hamilton-Jacobi equation for the surface recession and the heterogeneous reaction on the surface.
A numerical simulation of the coupling between the flow and the
surface recession is performed thanks to the open-source code OpenFoam, using a Reynolds Averaged Navier-Stokes (RANS) simulation and the two-equation k ? ? SST turbulence model. The flow is solved with a finite-volume (FV) scheme, and the evolution of the surface with a level-set method.

Topic: 1 - Computational modeling, simulation and design of new materials and processes
Abstract no. 1877

# Modelling accelerated testing conditions for SiC fibers accounting for different degradation mechanisms.

Presenting author: Dr BARANGER Emmanuel
Affiliation: LMT, ENS Paris-Saclay, CNRS, Université Paris-Saclay
61 avenue du président Wilson,94235 Cachan (France)

Co-authors:

SiC/SiC composites are good candidates for hot parts of engines in the aeronautic industry. In such parts, the loading is complex due to the mechanical stress state as well as environmental conditions. In this context and for long lifetimes, the experimental approach can become very expensive. Therefore, the definition of testing conditions that would lead to shorter testing times and respect the degradation scenario is an interesting approach, at least for validation. In case of multiple competing mechanisms, the definition of such conditions is not straightforward. In this paper, a methodology based on a fine physical modelling is proposed and applied to the oxidizing of SiC fibers.
For SiC fibers subjected to oxidizing atmosphere, different degradation mechanisms are involved. Parthasarathy has modelled them in 2016 in a unique framework. The strength of the fiber is related to the competing growth of grains, defects and of the formation of an external scale. This scale protects the fiber in some sense limiting the access of oxygen but can suffer from volatilization especially if water vapour is present.
The proposed strategy aims at preserving the evolution of the state variables describing the system (via a physical model). Starting from normal loading conditions, it is then possible to preserve the dynamics of the evolution of this state by adjusting loading conditions in time. A limit acceleration factor can be determined and a sensitivity analysis can give some information on the robustness of the method. The interest and drawbacks of such a method will also be discussed.

Topic: 1 - Computational modeling, simulation and design of new materials and processes
Abstract no. 2236

# COMPARISON OF MESO AND MACRO SCALE MODELLING OF COMPLEX 3D WOVEN COMPOSITE


Presenting author: M BéNéZECH Jean
Affiliation: LCTS - Safran Ceramics
3 allée de la Boétie,33600 Pessac (France)
Co-authors: COUéGNAT Guillaume (LCTS)



A prediction of the thermo-mechanical behavior of ceramics matrix composite necessitates a knowledge of their inner structure because of their complex multi-scale heterogeneities.
We proposed an original method allowed to build realistic models, as they are based on X-ray computed tomography of the material and a prior geometric model.
These resulting models are compatible directly with further numerical simulations at the mesoscopic scale.
They can be used as numerical twin to investigate in situ test for failure mechanism analysis.
However, this models accuracy requires too many degrees of freedom to be applied on a structural scale part.
Yet, an approximate macro-scale description may be sufficient to replicate the elastic behavior or first damage events.
Therefore, we established a macro-modelling framework, consisting in enriching macro-elements with the local composition.
It is achieved by implicitly consider information derived from the segmentation, i.e. local anisotropy (for yarns) and density of matter, through the constitutive equation of an equivalent laminate material.
Free of explicitly meshed each yarns, elastic and damage simulations are much more efficient and computationally achievable on structural scale parts.
Finite element simulations of the crossing weaving composite opening are then perform on both meso and macro models.
This study focused on investigating the nature and the amount of mesoscopic details necessary to simulate the elastic or even damage behavior, using the fully mesoscopic model as a reference.

Topic: 1 - Computational modeling, simulation and design of new materials and processes
Abstract no. 1849

# Thermodynamic stability of BaLn2O4 and phase relationship of the BaO - Ln2O3 system (Ln = La, Nd, Sm)

Presenting author: Prof GONG Weiping
Affiliation: Laboratory of Electronic Functional Materials, Huizhou University
46 Yanda Road, Huizhou University,Huicheng,516007 Huizhou (Guangdong, China)
Co-authors: SONG Jinxiang (Laboratory of Electronic Functional Materials Huizhou University), NAVROTSKY Alexandra (Peter A. Rock Thermochemistry Laboratory and NEAT ORU University of California Davis)

Alkaline earth, titanium and rare earth (Ln) metal oxides exhibit basic properties and may react at moderate temperatures at atmosphere to form ternary or quaternary oxides, for example, BaLn2O4 and Ba6-3xLn8+2xTi18O54, which have interesting structural and complicated physical properties, such as low temperature magnetic properties and microwave dielectric properties. Such materials are typically synthesized or find practical applications, and extensive work had been reported in the literature on the determination of physical properties. Hence, it is important to have accurate data on the formation and thermal stability. To facilitate such investigation on the BaO-Ln2O3-TiO2 system, reliable thermodynamic data on the ternary and quaternary phase are necessary. As a part of an ongoing program of Gibbs energy determination of ternary oxides related to magnetic phases in the BaO-Ln2O3-TiO2 system, the present investigation on the thermodynamic properties of BaLn2O4 (Ln = La, Nd, Sm, Gd) compounds were undertaken. On this basis, the local databases and phase diagram of the BaO–Ln2O3 system were set up by Calphad method. This work is a prerequisite for accurate predictions of the relationships among the composition, temperature and microstructure of complex functional materials containing BaO and Ln2O3.

Topic: 1 - Computational modeling, simulation and design of new materials and processes
Abstract no. 1873

# Modeling Defect Behavior in Perovskite Oxides and Their Superlattices


Presenting author: Prof XU Haixuan
Affiliation: The University of Tennessee Knoxville
336 Ferris Hall,Department of Mat. Sci. & Eng.,37996 Knoxville (TN, United States)
Co-authors:



This presentation focusing on modeling and simulations of point defect, particularly the oxygen vacancy in perovskite oxides and oxides superlattices using density functional theory (DFT) in combination with kinetic Monte Carlo (KMC) simulations. The effects of structural distortions, including octahedral rotation, polar modes, and interfacial effects on the oxygen vacancy formation energies will be discussed. The defect trajectories obtained from KMC simulations reveal that oxygen transport properties can be controlled by tuning the stacking period and sequence in the oxide superlattices. The KMC simulations also show that the dominant vacancy position may vary in the superlattices, depending on the superlattice structure and stacking period, contradicting the common assumption that point defects reside at interfaces. These results provide basic insights needed to control defect properties and behavior for technological applications.

Topic: 1 - Computational modeling, simulation and design of new materials and processes
Abstract no. 1970

# Multi-scale study of SiC-SiC / MI material and its application to aeronautical part

Presenting author: Dr DENNEULIN Sébastien
Affiliation: SAFRAN CERAMICS
105 avenue Marcel Dassault,33700 Mérignac (France)

Co-authors:

The introduction of Ceramics Matrix Composite (CMC) in hot part of aeronautical engines has become a reality on LEAP engines. In this context, SAFRAN develops CMC technologies such as 3D reinforced SiC/SiC composites obtained by CVI and Melt Infiltration. One of SAFRAN's challenges is to achieve the best compromise between improving material performances and designing composite structures.
In order to understand the thermo-mechanical behavior of these materials and parts (Fig. 1), it is necessary to understand existing links between their architecture at different scales and their global and local thermo-mechanical behavior [1]. These multi-scale studies allow to define multiaxial criteria of damage but also to optimize the architecture of the material and parts.
To do so, it is necessary to develop new methods based on the description of the material (tomography, statistical morphological studies, …), modeling (linear and nonlinear) and validation by testing (ex-situ and in-situ). SAFRAN is developing internally and with its academic partners, digital and experimental tools to meet these challenges.
The relevance of multi-scale approaches (modeling and tests) for the CMC materials has been demonstrated on the elastic behavior via several works at the RVE scale [2, 3]. The objectives are, on the one hand, to make these tools more robust so that they can be used by designers on parts [4], and on the other hand to extend their area of validity to non-linear behaviors (damage, creep, ...) [5].

Topic: 1 - Computational modeling, simulation and design of new materials and processes
Abstract no. 2235

# Experimental Study and Numerical Modeling of Pulse Flow CVI Process for Production of Organomorphic C/SiC Composites from Methylsilane

Presenting author: Prof KULIK Victor
Affiliation: Baltic State Technical University "VOENMEH"
1st Krasnoarmeiskaya st., 1,198005 Saint Petersburg (Russia)
Co-authors: BOGACHEV Evgeny (JSC Kompozit)

Chemical Vapor Infiltration (CVI) is one of the most efficient methods for production of high-quality Ceramic Matrix Composites (CMC). To fabricate CMC with SiC matrix, different precursors are currently used. Methylsilane (MS, CH3SiH3) is now considered as a promising alternative to haloid precursors. Principal advantages of MS are environmental safety of the process due to absence of chlorine and relative simplicity of the gas phase composition. For this reason, use of MS simplifies the hardware design of CVI reactors. Most profit of using MS as a precursor can be made in case of Pulse Flow CVI (PCVI). This modification of CVI is considered to be promising in comparison with traditional flow-through versions of CVI for densification of recently developed reinforcing preforms of organomorphic composites with small homogeneous pores (from several microns to tens of microns). One of the most important problems in CVI research and development is finding the optimal technological parameters providing minimal infiltration time and required quality. A possible way of solving this problem is in combination of experimental study and numerical modeling. In the present work, a 1D model of PCVI process for production of SiC matrix composites from MS precursor was developed. The mathematical model of the preform densification includes description of gas mixture transport inside the pores, SiC deposition, and evolution of the porous medium. Densification of the carbon reinforcing preform Ipresscon® was studied using this model. Variation of the material density and its distribution over the preform bulk in long-term PCVI process and effect of operating conditions on the preform evolution were analyzed in this work. The simulations were compared with experimental observations.

Topic: 1 - Computational modeling, simulation and design of new materials and processes
Abstract no. 1939

# SiC/SiC composites crack modeling at the fiber scale using a phasefield approach


Presenting author: M JAYET Thomas-david
Affiliation: Safran Ceramics
3 place jacques carat,94230 cachan (France)
Co-authors: BARANGER Emmanuel (CNRS)



SiC/SiC ceramic matrix composites developed for civil aviation have to replace certain metallic parts in the hot sections of aircraft engines. Such materials must therefore withstand many thermal loading cycles in order to limit the maintenance of the equipment and the replacement of parts. Many manufacturing processes have been developed and grant different mechanical and thermal properties to the matrix. In order to optimize manufacturing processes and obtain the best material attributes of parts regarding their environment, it is necessary to get a better understanding of the degradation mechanisms that occur at high temperatures. As the first damages occur at the fiber scale due to interphase and matrix
deposition, a detailed description of the microstructure is mandatory.
Several phases coexist at the scale of the fiber : silicon carbide fibers, a boron nitride or pyrocarbon interphase, a heterogeneous matrix and porosities. As a consequence such microstructure is very rich, which makes its finite element modeling a challenge. To develop a tool that helps to understand the behaviour of the material, a robust numerical strategy must be chosen to take into account the degradation phenomena within.
The method suggested here consists in implementing a di usive crack model based on a gradient-type damage model using a commercial code. Various formulations can be found from the literature but the emphasis will be put on a non-intrusive implementation of a damage law, regarding to the phase-field model. The damage tends to follow the Griffith model and adds the possibility of initiation, propagation and deflection of cracks without
mesh size dependency if elements are small enough. New numerical investigations will be conducted : a fiber scale 3D microstructure damaging due to post-manufacturing cooling and a crack deflection along a fiber submitted to a tensile stress will be presented.

Topic: 1 - Computational modeling, simulation and design of new materials and processes
Abstract no. 1896

# Effects of fiber and interfacial properties on fiber fragmentation and pullout in SiC-BN-SiC minicomposites


Presenting author: M CALLAWAY Evan
Affiliation: University of California, Santa Barbara
Materials Department,Engineering II Building 1355 UCSB,93106-5050 Santa Barbara (CA, United States)
Co-authors: ZOK Frank (University of California Santa Barbara)



Mechanical response of CMCs is critically dependent on properties of fibers and interfacial coatings. In the present study, we examine effects of key constituent properties on unidirectional composite response and fiber fragmentation via Monte Carlo simulations and tensile testing of SiC-BN-SiC minicomposites. A new model is developed for the distribution in fiber pullout length. Early fiber fractures occur in a random manner in accordance with the solution for a single fiber composite. Near the maximum load, fiber fractures are localized to the impending fracture plane, and the solution reverts to that of the single matrix crack problem. A high fidelity method for estimating in-situ fiber Weibull parameters is developed, and these values are compared to those obtained from fiber bundle tests. Differences in properties yield insights into degradation during composite processing and the degree of global load sharing during tensile testing.

Topic: 1 - Computational modeling, simulation and design of new materials and processes
Abstract no. 2293

# Novel bi-phasic numerical approach for cracking and delamination of carbon-carbon composites

Presenting author: Prof AIROLDI Alessandro
Affiliation: Dept. of Aerospace Science and Technology
Via La Masa 34,20156 MILANO (MILANO, Italy)
Co-authors: MIRANI Chiara (Dept. of Aerospace Science and Technology)

Carbon-carbon composite laminates are used in different engineering fields where structural efficiency and high temperature applicability are fundamental requirements. Low density and good material properties at high temperature make them suitable not only for thermal protection systems, but also for components with more significant structural roles in the aerospace field. However, the prediction of the structural integrity of carbon-carbon composite is complicated by multiple damage and failure modes, often characterized by a pseudo-plastic response, which is provided by fibre-matrix interactions, including fibre bridging phenomena, and internal friction.
The paper presents an innovative finite element approach, based on a bi-phasic decomposition of carbon/carbon needled laminates into idealized fibre and matrix phases. Fibre phase, representing the effect of the reinforcement continuity, is modelled by a mesh of membrane elements which are embedded into a mesh of solid hexahedral elements, modelling the matrix phase and matrix-dominated response. The non-linear constitutive law of the matrix model both transverse cracking and delamination, by means of a continuum damage mechanics approach, with damage evolution laws shaped according to cohesive zone models.
The delamination strength and toughness are characterized by means of an experimental protocol based on modified short beam tests, double cantilever tests and end-notched tests. The ability of representing matrix cracking is assessed by considering tensile tests on multi-directional laminates, with several lamination sequences. A coupling between interlaminar and intralaminar damage modes is introduced in the matrix law, exploiting the unique standpoint provided by the bi-phasic modelling approach adopted. Such coupling and the statistical distribution of defects in the matrix elements represent fundamental ingredients to model successfully the occurrence of localized failure, characterized by differently oriented cracks in the laminates, thus providing a model capable to representing realistically the complex damage pattern and the fracture response of the carbon/carbon laminates.

Topic: 1 - Computational modeling, simulation and design of new materials and processes
Abstract no. 1924

# Prediction of damage initiation and evolution in SiC/SiC composites by image-based modelling and FFT solver

Presenting author: Dr CHEN Yang
Affiliation: University of Oxford, Department of Materials
Parks Road,OX1 3PH Oxford (United Kingdom)

Co-authors: MARROW James (University of Oxford Department of Materials), GéLéBART Lionel (DEN-Service de Recherches Métallurgiques Appliquées CEA Université Paris-Saclay France), CHATEAU Camille (Laboratoire Navier UMR 8205 CNRS ENPC IFSTTAR Université Paris-Est France), BORNERT Michel (Laboratoire Navier UMR 8205 CNRS ENPC IFSTTAR Université Paris-Est France)

SiC/SiC composites are considered as promising candidate materials for high temperature applications, such as fuel cladding tubes in fission nuclear plants (i.e. accident tolerant fuel clad for current light water reactors or future high temperature gas-cooled reactors) or as structural components in fusion energy plants. This category of ceramic matrix composites exhibits user-designable thermomechanical properties that are closely related to the microstructures obtained by their manufacture. With a user-controlled porosity, these materials usually exhibit pseudo-ductile mechanical behaviour, which is the macroscopic consequence of micro-cracking within their multiscale structure. Therefore, a thorough understanding of micro-macro relationship is essential in order to apply SiC/SiC composites at the real industrial scale.
The complexity of their microstructures makes it necessary to investigate these materials using volume characterisation approaches. X-ray computed tomography provides 3D information on the bulk material, which allows numerical models to be generated with realistic microstructures. FFT solvers (or spectral methods) have been increasingly used in the context of image-based modelling, by virtue of their simplicity of mesh generation and efficiency of parallel implementation. We will present an FFT solver (available within the massively parallel code AMITEX) for predicting both the elastic response and the damage evolution of SiC/SiC composites. An in-situ tomography study allows the numerical predictions to be compared with or/and enriched by the experimental observations of cracks within the material.
This work has been partially supported by the EC Horizon 2020 project IL TROVATORE (grant 740415).

Topic: 1 - Computational modeling, simulation and design of new materials and processes
Abstract no. 2266

# Research Framework for Microstructure Evolution of Silicon Carbide


Presenting author: M MUñOZ Elias J.
Affiliation: Texas A&M University
575 Ross St,77840 College Station (TX, United States)

Co-authors: MARTINEZ Marco (Texas A&M University), ATTARI Vahid (Texas A&M University), DICKERSON Matthew (US Air Force Research Laboratory), RADOVIC Miladin (Texas A&M University), ARROYAVE Raymundo (Texas A&M University)



Advancing the cutting edge of aerospace technology requires the development of more powerful propulsion systems and by extension, structural materials that can reliably perform at the correspondingly more extreme operating conditions. SiC/SiC ceramic matrix composites present an attractive option, but maximum operating conditions are plagued by the existence of the relatively low melting point of residual silicon. Residual silicon remaining in the microstructure of reactive melt infiltrated ceramic matrix composites is a problem that hinders the full potential of CMC technology in high-temperature applications. Successful elimination of the residual Si phase can be accomplished by carefully alloying the Si infiltrant, thereby introducing a refractory intermetallic phase capable of consuming otherwise unreacted Si. Doing so, however, requires a comprehensive understanding of the evolution of the microstructure which can be approximated as the interface between a molten Si-X alloy and solid graphite. This project seeks to develop a framework by which the interaction of the available processing parameters of these composites influences the kinetic evolution of the composite via phase-field modeling. By efficiently sampling from the design space derived from major CMC processing parameters. Development of a physics based model that will end up being validated by experiments. We will be able to obtain a parameter-response as well as parameter-parameter relationships and interactions. The model will continually adapt to new results in order to help aid in materials design for CMCs.

Topic: 1 - Computational modeling, simulation and design of new materials and processes
Abstract no. 2317

# Empirically Derived Model for Alloyed Silicon Carbide Microstructural Evolution


Presenting author: M MARTINEZ Marco
Affiliation: Department of Materials Science and Engineering, Texas A&M University
1550 Crescent Pointe Pkwy, Apt 6207,77845 College Station (Texas, United States)
Co-authors: MUNOZ Elias (Department of Materials Science and Engineering Texas A&M University), MATTHEW Dickerson (US Air Force Research Laboratory), ARROYAVE Raymundo (Department of Materials Science and Engineering Texas A&M University), RADOVIC Miladin (Department of Materials Science and Engineering Texas A&M University)



SiC/SiC ceramic matrix composites prepared by reactive melt infiltration (RMI) offer an unparalleled combination of advantages for a variety of applications as a high-temperature structural material. Though the presence of Si in the CMC matrix offers certain advantages, it also limits the maximum operating temperature of RMI-prepared materials. Alloyed silicon infiltrants have proven effective in eliminating this residual silicon phase under specific processing conditions. Appropriately chosen, certain Si-X alloys offer the dual benefits of keeping required manufacturing temperatures low while providing a refractory silicide phase that consumes unreacted silicon. The nature of RMI involves a unique competition between the kinetics of the silicon carbide forming reaction and the fluid dynamics of the infiltration front capillary flow. Consequently, understanding the reaction kinetic response to changes in composite processing parameters is paramount to effective exploitation of infiltrant alloying. Developing a model that comprehensively captures this response will enable composite synthesis absent of any residual Si phase with simultaneously complete infiltration. While efforts have been made to study the microstructural evolution of individual Si-C-X systems, the problem at hand will benefit from a broader systems level approach. The use of statistical techniques will allow for the creation of an efficient and economical body of experimental data queried from an expansive design space. This dataset will be able to capture the microstructural response to individual parameters as well as higher order interactions among the parameters themselves. Regression analysis performed using machine learning techniques will then provide the means for an empirically derived model relating final Si-C-X microstructure to processing degrees of freedom such as alloy composition, reaction time, and temperature.

Topic: 1 - Computational modeling, simulation and design of new materials and processes
Abstract no. 2119

# Phase evolution of ternary W-C-Co carbides depending on temperature and activity of the elements

Presenting author: Dr KIM Jiwoong
Affiliation: Korea Institute of Geoscience and Mineral Resources
Gajeong-ro,Yuseong-gu,34132 Daejeon (South Korea)

Co-authors: MYUNGJAE Kim (Mineral Resources Research Division Korea Institute of Geoscience and Mineral Resources 124 Gwahak-ro Yuseong-gu Daejeon 34132 Republic of Korea), YONGSEON Kim (Inha University Materials Science and Engineering Incheon 22212 Republic of Korea), HANJUNG Kwon (Mineral Resources Research Division Korea Institute of Geoscience and Mineral Resources 124 Gwahak-ro Yuseong-gu Daejeon 34132 Republic of Korea)

We investigated phase evolution of ternary W-Co-C carbides according to the phase stability change depending on temperature and activity of the elements. To obtain the stability, we employed ab initio thermodynamic calculations, whose reliability was ensured with experimental evidence for the phase evolution. Temperature effects calculated from phonon vibration analyses within quasi-harmonic approximation indicated that the stable phase area of W4Co2C was reduced with increasing temperature but its position moved gradually closer to either pure carbon or cobalt (a_C=a_Co=1). This is the main reason why W4Co2C phase has been frequently observed in W-Co-C system. In addition, it is difficult to observe W3Co3C due to its position of the stable phase. This research will provide fundamental information for ternary W-Co-C carbides.

Topic: 1 - Computational modeling, simulation and design of new materials and processes
Abstract no. 2776

# Ceramic Matrix Composites (CMCs): Manufacturing and Microstructural Effects on the Mechanical Properties using the Parametric HFGMC


Presenting author: M YANNAY OMRI
Affiliation: Tel-Aviv University & RAFAEL
Ginat egoz 4,3708458 Pardes Hana (Israel)
Co-authors: KORESH Ido (RAFAEL), SHEKHTER Pini (RAFAEL), PADAN Royi (RAFAEL), ABOUDI Jacob (Tel-Aviv University), HAJ-ALI Rami (Tel-Aviv University)



Advanced Carbon-based Ceramic Matrix Composites (CMCs) are important in today's aviation industry because of their unique properties - can withstand high temperature and severe erosion conditions, while maintaining the composites strength at relatively lower weight.
However, these unique properties depend on the microstructure of the formed material through the CMC's production process. The use of refined micromechanical methods, such as the parametric High Fidelity Method of Cells (HFGMC) is crucial in order predict the overall thermo-mechanical properties and how they are related to the optimal ratio of the phases, towards improving the desired and objective properties. Furthermore, applying this new micro-scale analysis can save time and money by replacing the experiments on such expensive material system. It can even generate added values that one cannot extrapolate in standard experimental approach such as predicting the overall anisotropic mechanical properties, and the stress states at the micro scales. It should be noted that all inputs for the proposed micromechanical simulations can be easily obtained by using basic physical measurements combined with data in the open literature, such as material's microstructure and phase's properties.
This research presents a new framework for prediction the overall thermo-mechanical properties of CMCs using the parametric HFGMC starting from the manufacturing process of CMCs by Liquid Silicon Infiltration (LSI) method. For each production stage, a Repeated Unit Cell (RUC) model is applied in order to achieve more reliable results at each production level. The proposed micromodels are nested in a multi scale analysis in order to generate the overall effective properties of the CMC. Finally, the effects of material microstructural features on the overall elastic properties are investigated, reported and discussed.

Topic: 1 - Computational modeling, simulation and design of new materials and processes
Abstract no. 2053

# Axisymmetric and 3-D Numerical Simulations of Thermomechanical Behavior during the Spark Plasma Sintering "SPS" Process of Polycrystalline Materials

Presenting author: Dr MECHIGHEL Farid
Affiliation: University of Annaba
Annaba,Annaba,23000 Annaba (Algeria)

Co-authors: PATEYRON Bernard (University of Limoges), EL GANAOUI Mohammed (UIT Longwy - University of Loraine)

Spark Plasma Sintering (SPS) is a promising rapid consolidation technique that allows a better understanding and manipulating of sintering kinetics and therefore makes it possible to obtain polycrystalline materials (ceramic or metallic) with tailored microstructures. A numerical simulation of the electrical, thermal and mechanical coupling during SPS is performed. Equations for conservation for energy, electrical charge and mechanical equilibrium are solved simultaneously. The strong coupling in term of temperature, on the thermal conductivity and electrical resistivity are considered. The effect of lateral surfaces radiation on the overall heat transfer in the SPS system is also considered.

Topic: 1 - Computational modeling, simulation and design of new materials and processes
Abstract no. 2052

# Micromechanically motivated damage modelling of ceramic matrix composites

Presenting author: Dr FINDEISEN Claudio
Affiliation: Fraunhofer IWM
Woehlerstraße 11,79108 Freiburg (Germany)

Co-authors: HOHE Jörg (Fraunhofer IWM)

The non-brittle, damage resistant effective material behaviour of ceramic matrix composites (CMCs) is a necessary condition for the industrial application of CMCs in structural components. For the design of such components a reliable damage model is required. Such a model should be able to describe various effects that can be easily observed from simple tension and compression experiments. These effects include the anisotropic damage initiation and anisotropic damage evolution, transverse damage effects, the damage deactivation under compression and residual (plastic) strains. However a good model should also lead to qualitatively meaningful results for stress states that cannot be validated experimentally. For the latter requirement it is helpful to formulate a model based on micromechanical mechanisms. This poster gives an overview on the recent activities at Fraunhofer IWM to formulate, validate and implement such a micromechanically motivated model. The model is based on the damage effect of cracks orientated at different angles and formulated using the Gibbs free energy potential. Using the Gibbs potential leads to a formulation in stress space that facilitates the model calibration and validation however requires more effort in the implementation. The model capabilities describing the damage behaviour of woven SiC/SiC composites as well as aspects of the implementation will be presented and discussed.

# Topic: 2

# Fibers and preforms

Topic: 2 - Fibers and preforms
Abstract no. 1993

# Environmental Effects on SiC and Oxide Fiber Mechanical Properties

Presenting author: Dr HAY Randall
Affiliation: USAF Materials and Manufacturing Directorate
Bldg 655 Area B,WPAFB,45433 Dayton (OH, United States)

Co-authors:

The environment affects CMC mechanical properties at intermediate and high temperatures. Mechanistic understanding of these effects requires knowledge of mechanisms that affect fiber properties. At least five kinds of mechanisms may operate: SiC Fibers 1) Increases or decreases in fiber strength related to the residual stresses that develop in scales formed during SiC fiber oxidation in various environments. SiC and Oxide Fibers 2) Subcritical crack growth in both SiC and oxide fibers facilitated by surface active gas species in various environments at intermediate and high temperatures. 3) Degradation associated with grain boundary wetting of a transient liquid phase. 4) Degradation by selective reaction of major or minor fiber phases with environment components. 5) Degradation associated with enhanced fiber grain growth in some environments.

More than one mechanism may simultaneously operate, and in some cases mechanisms may be synergistic. Evidence for the various mechanisms is reviewed and discussed. Data that has been collected and further data necessary for modeling and quantification of the various effects is presented. This data includes information like oxygen permeation rates in glass and crystalline SiO2 in various environments, the effects of environment on SiO2 crystallization rates, and the effects of impurities on all these processes. Other necessary data and information includes identities of surface active species, wetting phases, deleterious minor fiber phases and impurities, and mechanisms that enhance grain growth.

Topic: 2 - Fibers and preforms
Abstract no. 2246

# High temperature oxidation of SiC fiber-reinforced type CMCs


Presenting author: Dr SHIMODA Kazuya
Affiliation: National Institute for Materials Science
1-2-1,Sengen,3050047 Tsukuba (Ibaraki, Japan)
Co-authors: KONDO Sosuke (Tohoku University), HINOKI Tatsuya (Kyoto University)



SiC fiber-reinforced type ceramic matric composites (CMCs) are one of very attractive engineering materials in particular for advanced nuclear use due to their outstanding properties of excellent high temperature strength, relatively high thermal conductivity and exceptional low-radioactivity. It is proposed to replace the current zirconium-based fuel cladding in light water reactors with the CMCs, which significantly provide more oxidation-resistant enhancing safety margins during severe accidents. The CMCs basically require weak fiber/matrix interphase like pyrocarbon to avoid catastrophic fracture manner, but the interphase material and its thickness are keys to determine material properties and serviceability limit against oxidation, which degrade with the increase of temperature above 500 oC. This present work starts from fabrication technology of novel CMCs developed in Japan, and then evaluates high temperature performance especially in terms of oxidation resistance below 1500 oC, characterized by weight change using thermo-gravimetry differential thermal analysis (TG-DTA), microstructural observations using field emission scanning electron microscopy (FE-SEM) with energy dispersive x-ray spectroscopy (EDS), gas analysis using photoionization mass spectrometry (PIMS), and mechanical properties. It was confirmed that the oxidation resistance of CMCs had the strong connection with/without interphase between fibers and matrix as well as interphase thickness. Also, many options for environmental barrier coatings for the CMCs were investigated. Those results are provided and discussed especially in comparison to representative heat-resistant alloys, such as stainless steels, nickel-base and zirconium-base alloys.

Topic: 2 - Fibers and preforms
Abstract no. 2381

# Carbon fibers - structure and mechanical properties at extreme conditions

Presenting author: Prof PETERLIK Herwig
Affiliation: University of Vienna, Faculty of Physics
Boltzmanngasse 5,1090 Vienna (Austria)

Co-authors:

Carbon fibers are the most important reinforcing material for light-weight composites, ranging from moderate temperatures applications such as airplanes or bicycles to extremely challenging high temperature materials as used for rocket nozzles or wing edges. One of the surprising features of carbon fiber reinforced materials is that a number of mechanical properties are improved at high temperatures, which is shown here as an example for Young's and shear moduli of C/C measured by resonant ultrasound spectroscopy. The structural origin of this improvement is that fibers from polyacrylonitrile precursor have basic structural units consisting of graphene planes in a turbostratic configuration, which are rearranged under exposure to combined temperature and load. This dynamic change of the structure can be avoided by a suited high temperature treatment without load. In-situ experiments using a micro- and nanobeam from synchrotron radiation sources allow following the structural development up to temperatures of 2000 oC directly, even for single carbon fibers with a diameter of only some microns.

Topic: 2 - Fibers and preforms
Abstract no. 2033

# Comparative Thermostructural Properties of Commercial and Pre-Commercial Silicon Carbide Fibers


Presenting author: Dr SHAY Harrison
Affiliation: Free Form Fibers
10 Cady Hill Boulevard,12866 Saratoga Springs (NY, United States)

Co-authors: HARRISON Shay L. (Free Form Fibers), SHIRVAN Koroush (Massachusetts Institute of Technology)



Commercially available silicon carbide were tested in parallel with pre-commercial ones for: (1) Room and high (1500ºC) temperature tensile properties, (2) Tensile creep at 1315ºC and 1500ºC, (3) Oxidation resistance in air at 1600ºC, (4) Dissolution in pressurized water reactor operating conditions, and (5) Gamma ray exposure. This talk presents a compendium of experiment designs and their results collected in the course of a two-year campaign of evaluation.

Topic: 2 - Fibers and preforms
Abstract no. 1963

# Direct comparison between multi-filament tow and monofilament tensile test results of SiC fibers


Presenting author: Prof OGASAWARA Toshio
Affiliation: Tokyo University of Agriculture and Technology
2-24-16, Naka-cho,184-8588 Koganei-shi (Tokyo, Japan)
Co-authors: TAKUYA Aoki (Japan Aerospace Exploration Agency (JAXA), ICHIKAWA Hiroshi (Advanced Institute of Materials Science), YAMAOKA Hiroyuki (Ube Industries Ltd.), OKAMURA Mitsuyasu (NGS Advanced Fiber Co. Ltd.), OKUIUMI Shin-ichi (Tokyo University of Agriculture and Technology)



Multifilament tow tensile test method is attractive because it is possible to obtain the stochastic fiber strength data with lower effort compared with monofilament tensile test method. However, the multifilament tow test method has not been verified sufficiently. This study attempted to make direct comparison between multi-filament tow and monofilament tensile test results. Four kinds of SiC fibers, Nicalon (NL-201), Hi-Nicalon, Hi-Nicalon Type-S, Tyranno ZMI were evaluated. Multi-filament tow tests were conducted in accordance with EN1007-5: 2010 (European committee for standardization). The gauge length was 200 mm. Effects of displacement rate, and lubricant application on the experimental results were examined. During multi-filament tow tensile tests, unstable fiber breakage behavior, i.e. sudden drop in the tensile load, was often observed. Lower loading rate was effective to make the fiber breakage behavior stable, but unstable failure still occurred for Hi-Nicalon fiber. Sizing agent (PVA) seemed to affect the unstable failure. Glycerol as lubricant was dripped on multifilament tow using a syringe before tensile testing. It seems to be effective to make the failure stable, because of reduction of friction and/or dampening stress waves.
Monofilament tests were made in accordance with ISO 19630:2017. The gauge length was 25 mm. The tensile strength distribution data obtained by multi-filament tow testing didn't show good agreement with those obtained by monofilament tests. Generally, the Weibull's shape parameters, m, obtained by multi-filament tow tests were relatively higher, whereas the shape parameters, s0 were lower in spite of the consideration of the gauge length effect. The validity of multifilament tow test method could not be experimentally demonstrated in this work. Further experimental and analytical studies are required for establish the multi-filament tow tensile test method.

Topic: 2 - Fibers and preforms
Abstract no. 2021

# Properties of Continuous Alumina & Mullite Fibers „Made in Germany“ and resulting CMCs

Presenting author: M KLUTHE Christopher
Affiliation: Rath GmbH
Krefelder Straße 680,41066 Mönchengladbach (Germany)
Co-authors: FINKE Vera (Rath GmbH), KANKA Bernhard (WPX Faserkeramik GmbH)

Continuous alumino & mullite ceramic fibers are produced by Sol-Gel processes. Developement and production of those fibers require unique chemical engineering.
Therefore the availability of those fibers is very limited. As an expert for alumino & mullite staple fibers derived from the Sol-Gel process, Rath (Mönchengladbach, Germany) took up the challange and developed their on type of continously spun oxide fibers. The properties and microstructure of those fibers shall be discussed in the first part of the presentation.
All-Oxide-ceramic-composites based on continuous alumino- or alumino-silicate (mullite) fibers and alumino and mullite matrices have been developed for over 20 years. Those composites exhibit exceptional damage tolerance and have a high bending strength and Young‘s modulus. Those properties, along with the outstanding thermal shock resistance of the composite, make it an excellent choice for components used in an oxidizing environment at high temperatures. In the second part of the the presentation mechanical properties and micro- and mesostructure of an oxide oxide ceramic matrix composite , which has been produced at the WPX Faserkeramik Company (Troisdorf, Germany), based on the new Rath-fiber shall be demonstrated.

Topic: 2 - Fibers and preforms
Abstract no. 1744

# The crystallization of the aligned spinning Si-C-N nanofibers via polymer-derived-ceramic method


Presenting author: Mme LI ZHAOCHEN
Affiliation: Northwestern Polytechnical University
127 West Youyi Road,Beilin District,710072 Xi'an (Shaanxi, China)
Co-authors: CHENG LAIFEI (Northwestern Polytechnical University), YE FANG (Northwestern Polytechnical University)



As a kind of one-dimensional Si-C-N materials, the Si-C-N aligned nanofibers have high degree of orientation, big aspect ratio and multi-phase composition, which may have the comprehensive performance on structure and function. In this paper, based on the combination of electrospinning and polymer-derived-ceramic methods, Si-C-N aligned nanofibers were prepared with polycarbosilane used as the polymer precursor and nitrogen as N source. After being shaped under the control of optimized spinning parameters, both the microstructure and chemical composition of the nanofibers were investigated to explore the effect of different heat-treated temperatures on the nanofibers' crystallization. When the temperature increases from 1300 °C to 1600 °C, amorphous SiC microcrystals and SiOxCyNz phase are formed first, and then Si3N4 grains gradually crystallize. The carbon phase covers the fibers as well as dispersedly embeds in the form of particles. The morphology of the carbon layer covering the fibers changes from amorphous to lamellar and vortex-like microstructure, with an increase in the degree of disorder. The prepared Si-C-N aligned nanofibers may contain SiC crystallites, Si3N4 crystallites, C phase and amorphous SiOxCyNz phase with many defects and heterogeneous interfaces.

Topic: 2 - Fibers and preforms
Abstract no. 1961

# Microstructure evolution and mechanical performance of oxide ceramic mini-composites after thermal exposures

Presenting author: Mme FARHANDI Hedieh
Affiliation: Bremen University
Am Biologischen Garten 2,IW 3, Room 2300,28359 Bremen (Germany)

Co-authors: ALMEIDA Renato (Bremen University), TUSHTEV Kamen (Bremen University), REZWAN Kurosch (Bremen Univeristy)

Microstructure evolution and mechanical performance of oxide ceramic mini-composites after thermal exposures
H. Farhandi, R. S.M. Almeida, K. Tushtev and K. Rezwan
Oxide ceramic matrix composites are materials with high strength and considerable toughness and are, most importantly, resistant to oxidizing environments. However, the fibers which are used as the reinforcement in the composites are susceptible to strength loss due to grain growth at temperatures above 1000°C. As processing and application of these materials are at high temperatures (T > 1000°C), the elucidation of the microstructural evolution and mechanical performance of the composites at elevated temperatures is essential. For this purpose, the mechanical properties and microstructure of mini-composites consisting of NextelTM 610 fibers embedded in alumina matrix with high purity were investigated. A rate controlled sintering technique of mini-composites was carried out to achieve the densification of the mini-composites while inhibiting the grain growth of the matrix at the final stage. For this purpose, a two-step sintering process was applied and the results were compared with the conventionally sintered mini-composites. The investigations showed promising results in terms of lower fiber degradation and higher composite strength. For better understanding this phenomena, stand alone fibers and matrix were fired in two-steps and the results were compared to further explain the higher strength of the mini-composites.

Topic: 2 - Fibers and preforms
Abstract no. 1851

# "OXCEFI" Oxide ceramic fibers and fabrics developed by DITF Denkendorf


Presenting author: M CLAUß Bernd
Affiliation: Deutsche Institute fuer Textil- und Faserforschung Denkendorf
Koerschtalstr. 26,73770 Denkendorf (Germany)

Co-authors: PFEIFER Stephanie (DITF Denkendorf), BAUDER Hans-Juergen (DITF Denkendorf), BUCHMEISER Michael R. (University of Stuttgart and DITF Denkendorf), KANKA Bernhard (WPX Faserkeramik GmbH Troisdorf Germany), PüTZ Monika (WPX Faserkeramik GmbH Troisdorf Germany), PRITZKOW Walter (Walter E.C. Pritzkow Spezialkeramik Filderstadt Germany)



Oxide fiber research and development has been started 30 years ago at the German Institutes of Textile and Fiber Research (DITF) in Denkendorf, Germany. In this time fundamental and applied research was conducted in order to come to high level oxide fibers based on alumina and mullite. This goal has been achieved and the talk shall give an overview about the developments and current data of the "OXCEFI fibers".
During the last years DITF has set up a complete pilot line for fiber production, which contains all steps from dope preparation over dry spinning to calcination and sintering. The line was designed to produce fiber quantities, which are large enough for being tested in ceramic matrix composites (OCMC) and for further developments. During 2018 also a high end jacquard weaving technology has been established at the institutes and woven fabrics from "OXCEFI fibers" have been successfully produced.
Oxide CMCs based on the DITF fibers and fabrics have been fabricated and tested by different companies with positive results. Material data will be presented in the talk.
Further developments at DITF are focused on doped oxide fibers and fibers with dispersion structures aiming at reduced grain growth and enhanced creep stability.

Topic: 2 - Fibers and preforms
Abstract no. 1743

# Synthesis and characterization of carbon-poor SiC nanowires via vapor-liquid-solid growth mechanism

Presenting author: Mme GUO Chuchu
Affiliation: Northwestern Polytechnical University
127 West Youyi Road,Beilin District,710072 Xi'an (Shaanxi, China)

Co-authors: CHENG Laifei (Northwestern Polytechnical University), YE Fang (Northwestern Polytechnical University)

With a wide range of applications, SiC nanomaterials are ideal materials for manufacturing high-temperature, high-voltage, high-frequency, low-energy, high-power, and anti-radiation devices. Among, SiC nanowires have a large specific surface area and excellent functionality. Vapor-liquid-solid growth mechanism leads to high-quality SiC nanowires.

Here, we report the synthesis and growth mechanism of 3C-SiC nanowires and SiC nanowires containing reduced amount of C, which are grown on single-crystal Si via pyrolysis of polycarbosilane (PCS) by adjusting pyrolysis temperature and precursor state. The synthesized SiC nanowires have a diameter of 50 nm. The SiC nanowires were grown to a thickness of 43.75 ?m. As the pyrolysis temperature increases from 1050 °C to 1550 °C, Ni deposited on the substrate first catalyzes the tortuous nanowires rich in carbon and carbon. Subsequently, the SiC nanowires were grown by a vapor-liquid-solid mechanism. The pyrolysis temperature of 1350 °C is most beneficial to the formation of carbon-poor SiC nanowires. The surface amorphous layer of the SiC nanowires formed from the solid PCS precursor is only 0.86 nm, which is due to the high temperature stability of the solid PCS precursor and the slow reaction rate after melting.

Topic: 2 - Fibers and preforms
Abstract no. 1994

# Creep properties of SiC fiber

Presenting author: M NAKAMURA Takeshi
Affiliation: IHI Corporation
229, Tonogaya,Mizuho-machi,190-1297 Nishitama-gun (Tokyo, Japan)
Co-authors: KOTANI Masahiro (IHI Corporation)

SiC/SiC has been developed for the high temperature application like turbine parts of aero-engine. Creep properties of several SiC fibers including sintering fibers like Hi-Nicalon type-S (NGS Advanced Fibers Co.,Ltd., ) and Tyranno SA (UBE Industries. LTD.) were compared by the mono-filament creep test at the high temerature over 1200 degree Celsius. Mono-filament creep test were done in the air. Creep strain rate of these fibers were measured with and without BN coating. BN coating was applied on these fibers by the continuous CVD process. Micro observation and X-ray diffraction before and after the test were also done to study the difference of the creep strain rate.

Topic: 2 - Fibers and preforms
Abstract no. 1727

# From lab to fab – development of oxide ceramic fibers at the Fraunhofer HTL


Presenting author: M RüDINGER Arne
Affiliation: Fraunhofer ISC / Zentrum HTL
Neunerplatz 2,97082 Würzburg (Bavaria, Germany)

Co-authors: HERBORN Ralf (Fraunhofer ISC Center for High Temperature Materials and Design HTL), NöTH Dr. Andreas (Fraunhofer ISC Center for High Temperature Materials and Design HTL)



In the last years the Fraunhofer ISC Center HTL worked on the development of endless multifilament oxide ceramic fibers. E.g., based on chlorine free aqueous precursors in the system Al2O3 – SiO2, the entire process route from spinning dope preparation to an endless sintering process was developed.  Starting with a few 100 ml spinning dope and a spinning nozzle for 20 filaments it was possible to process short fibers with a length of 0.2 m in batch thermal processes. The next step was an upscaling of all steps to 2 l of spinning dope per batch, 100 filament spinning nozzle, fiber lay-up in baskets as endless fibers with a length of 200 m, a batch calzination and endless sintering of one multifilament. Since 2017 the Fraunhofer HTL is building a fiber pilot plant for the manufacture of ceramic fibers in a larger scale. The capacity of the oxide ceramic fiber line will be about 1 – 3 t/year of a 500 – 1000 filament endless roving. The start of operation of the oxide fiber line is planned at the end of 2019. Current work addresses the build up of the new fiber line and the transfer of the fiber-spinning-process from today's lab-scale to the fab-scale of the fiber pilot plant.

Topic: 2 - Fibers and preforms
Abstract no. 1860

# Development of a method to characterize modulus and tensile strength of oxide fibre: comparison with the common method

Presenting author: Mme LEFEBVRE Marie
Affiliation: SAFRAN CERAMICS
rue de Touban,33185 Le Haillan (France)
Co-authors: HUGUET Chrystel (Safran Ceramics)

A tensile method has been developed to characterize in one way the tensile strength and the modulus for oxide fibre.
This method follows a standard method used for carbon and glass industries. The main challenge was to find the best preparation of the sample to perform the mechanical test: size of the tow, compatible resin, preparation of sample etc.
Statistical analysis have shown that this method is reproducible for different batches of fibres.
This method was compared to other standard method as single filament test.
Description of the method and comparative results will be discussed in this presentation.

Topic: 2 - Fibers and preforms
Abstract no. 1995

# Degradation behavior of SiC/SiC composites

Presenting author: M KAWANISHI Ken
Affiliation: IHI INC.
150 East 52nd Street, 24th Floor,10022 New York (NY, United States)

Co-authors: CALLAWAY Ben (Univeristy of California Santa Barbara), ZOK Francis (University of California Santa Barbara)

Oxidative degradation of SiC/SiC composites remains one of the most critical factors limiting the use of these composites in industrial parts, especially in water-containing environments. Here we present an evaluation of the properties and degradation mechanisms operative in SiC fibers in three forms: bare, coated with BN, and within unidirectional SiC/SiC mini-composites. The test conditions include temperatures and environments relevant to gas turbine applications. Microscopy and fractography are used to probe the degradation mechanisms. Additionally, in-situ constituent properties are inferred from measurements of hysteresis response. The key degradation mechanisms and mitigation strategies are discussed.

Topic: 2 - Fibers and preforms
Abstract no. 2031

# Micro-Trellis™: Novel non-woven 3D fabric architecture for CMC and Hybrid MMC-CMC

Presenting author: Dr PEGNA Joseph
Affiliation: Free Form Fibers
10 Cady Hill Boulevard,12866 Saratoga Springs (NY, United States)

Co-authors:

When additive manufacturing is applied to filamentary structures – as is the case with 1½-D Printing – it opens up opportunities for new 3-D fiber architectures that obviate the need for weaving or braiding. One such architecture is micro-trellis, which rely on ribbons of continuous laser printed Silicon Carbide fibers are layer over massive arrays of through thickness short Silicon Carbide fibers. This architecture offers a lightweighting alternative to woven fabrics or braided tubular structures; circumventing the strength degradation and porosity inherent to woven tow fabrics.
SiC-SiC composites with this fiber architecture were evaluated for thermal conductivity and flexural strength.
In one embodiment, the micro-trellis architecture allows the joining of metal and CMC into a hybrid MMC-CMC structure. This presentation also reports on joint characterization results to date.

Topic: 2 - Fibers and preforms
Abstract no. 1940

# The effects of weave bending angles on the integrity of Alumina fibres.

Presenting author: Mme VALENZUELA Erin
Affiliation: University of Birmingham
School of Metallurgy and Materials,Engineering and Physical Sciences,B15 2TT Edgbaston (Birmingham, United Kingdom)
Co-authors:

The demand for thermo-mechanical stability within Ceramic Matrix Composites (CMCs) is ever-growing within the aerospace and defence industry. There are many methods that can be used to solve this issue, one of rising note includes adding strength via additional fibres in different dimensions of the composite. Two dimensional CMCs are well known; adding half a dimension via a stitching method gives an additional direction of strength, and finally a concept known as Noobing: three-dimensional woven fibre preforms, strengthens in a third direction. This is a concept that has been recently explored within the realm of carbon fibre, however introducing alumina fibres to the process to help strengthen oxide/oxide CMCs is a new area of investigation. The experimental work will aid in an analysis into whether alumina fibres can withstand the bending stresses experienced within the weaving process. Being able to understand the characteristic bending strains associated with different weaves will enable the correct selection of fibres as well as an understanding into which weaving configurations work best for a more brittle fibre. This study will consider the radius of the fibres, the angles in which the weaves bend the fibres and the area in which these weave contortions take place. The analysis will cover experimental comparisons, Optical Microscopy and SEM to understand the impact of the bending angle on the fibres. This analysis will then be used to generate an understanding into how the currently available alumina fibres react under given stresses, how the weaving process impacts the integrity of the fibres, and whether the fibres of the CMC can really be the focus for the dimensional strengthening of such composites.

Topic: 2 - Fibers and preforms
Abstract no. 1951

# Fabrication of SiC fiber textiles via silicidation treatment of carbon fabrics with SiO gas


Presenting author: Dr ISTOMINA Elena
Affiliation: Institute of Chemistry, Komi Science Centre of the Ural Branch of the Russian Academy of Sciences
Pervomaiskaya, 48,167982 Syktyvkar (Russia)

Co-authors: ISTOMIN Pavel (Institute of Chemistry Komi Science Centre of the Ural Branch of the Russian Academy of Sciences), NADUTKIN Aleksandr (Institute of Chemistry Komi Science Centre of the Ural Branch of the Russian Academy of Sciences), GRASS Vladislav (Institute of Chemistry Komi Science Centre of the Ural Branch of the Russian Academy of Sciences)



Technical SiC textile materials are very attractive for use in a variety of applications including energy conversion, aerospace, and automotive systems, because of the superior mechanical properties, high thermal stability, and excellent chemical resistance of SiC fibers. Commercially available SiC fibers suitable for manufacturing textiles are currently produced through the pyrolysis of organosilicon precursor filaments. A novel alternative approach to the fabrication of SiC fiber textiles via silicidation treatment of carbon fabrics with SiO gas is presented herein. It is shown that the silicidation treatment above 1350 °C for several hours allows full conversion of carbon fiber precursors into SiC textile materials in accordance with the following reaction:

$2C + SiO = SiC + CO$.

It is shown that the choice of reactive source for SiO gas generating strongly influences the synthesis conditions that in turn affects the microstructure characteristics of SiC fibers. The better results are achieved when using equimolar powder mixture of Si and $SiO_2$ as SiO gas source. Original batch-type reactors of different design for the synthesis of SiC fiber textiles have been also developed.



This work was financially supported by the Russian Foundation for Basic Research (grant #18-08-01460).

Topic: 2 - Fibers and preforms
Abstract no. 2100

# Evaluation of mechanical behavior of fiber fabrics with soft matrix by bending recovery test


Presenting author: M KINUGASA Takahiko
Affiliation: Osaka Prefecture University
1-1 Gakuen-cho,Naka-ku,599-8531 Sakai (Osaka, Japan)

Co-authors: NARISAWA Masaki (Osaka Prefecture University), YAMAGATA Manabu (KITAMURA CHEMICALS CO. LTD.), SAITO Akiyoshi (Kawamura Gishi Co. Ltd), SUZUKI Junya (Kawamura Gishi Co. Ltd)



The mechanical behavior of 2D-fiber fabrics plates with soft matrix was evaluated by three-point bending recovery test. The matrix used in the composite was the epoxy resin, which can maintain softness even after curing. The reinforcement used in the composite was carbon fiber T-300 with 1000 monofilaments. These resin-fiber composites are named “Soft-CFRP”. The thickness of the tested composite plates was 1 mm, the span width was 48 mm, and the applied displacements were 2,4,6,8,10 mm. The radius of curvature just under the indenter was calculated to be in the range of 20-100 mm. From mechanical hysteresis curve obtained by the bending recovery test, load reduction rate, residual displacement rate, energy loss rate, and normalized repulsion force were defined. By analyzing these parameters, the characteristics of Soft-CFRP were quantitatively expressed. The “normalized repulsion force” of Soft-CFRP kept stability value even after deep bending, while that of ordinary plastics such as polypropylene and polyethylene suddenly fell after the deep bending. In addition, Soft-CFRP recovered to original shape smoothly after the test. “Residual displacement rate” was always below 10%. Such information given by analyzing the mechanical hysteresis curves of thin plate materials, like soft-CFRP, would be useful even in analyzing the production process of ceramic-matrix-composites (CMC), because matrix source of CMC is usually applied on the fiber woven as soft slurry-like materials. In addition, CMC production process is often accompanied by the process of piling up the thin plates and pressure application on the formed layers.

# Topic: 3

# Interfaces and interphases

Topic: 3 - Interfaces and interphases
Abstract no. 1974

# Rare-earth disilicate fiber coatings for SiC/SiC CMCs


Presenting author: Dr BOAKYE Emmanuel
Affiliation: UES Inc.
4401 Dayton-Xenia Road,45432 Dayton (Ohio, United States)

Co-authors: MOGILEVSKY Pavel (UES), PARTHASARATHY Triplicane (UES Inc.), KEY Thomas (UES Inc), CINIBULK Michael (Materials and Manufacturing Directorate Air Force Research Laboratory Wright-Patterson AFB), HAY Randall (Materials and Manufacturing Directorate Air Force Research Laboratory Wright-Patterson AFB), OPEKA Samuel (UES Inc.), CORNS Randall (UES Inc.)



Rare-earth disilicates (RE2Si2O7) are potential oxidation-resistant alternatives to carbon or BN fiber coatings for SiC/SiC CMCs. Our prior work experimentally demonstrated that rare earth disilicates may work as a weak interface in fiber-reinforced SiC/SiC CMCs. However, the effect of oxidation, especially in water vapor, on their functionality as a weak interface has not yet been tested. In this work, SiC/SiC minicomposites with Y2Si2O7 interface were exposed to steam and tensile tested. The effect of steam on the functionality of the Y2Si2O7 interface coatings was studied and will be reported.

Topic: 3 - Interfaces and interphases
Abstract no. 1846

# Optimization of the SiC/SiC composites fiber/matrix interfaces for nuclear applications

Presenting author: Dr BRAUN James
Affiliation: CEA Saclay
Bat 460 - p 33 - PC52,91191 Gif-sur-Yvette Cedex (France)

Co-authors: FELLAH Clémentine (Laboratoire de Géologie de Lyon Université de Lyon Ecole Normale Supérieure de Lyon Université Claude Bernard Lyon 1 CNRS 46 Allée d'Italie 69364 Lyon Cedex 7 France), BUET Emilien (Deceased), SAUDER Cédric (DEN-Service de Recherches Métallurgiques Appliquées CEA Université Paris-Saclay F91191 Gif-sur-Yvette France), LORRETTE Christophe (DEN-Service de Recherches Métallurgiques Appliquées CEA Université Paris-Saclay F91191 Gif-sur-Yvette France), BERGER Marie-Hélène (MINES ParisTech PSL Research University MAT - Centre des matériaux CNRS UMR 7633 BP 87 91003 Evry France)

SiC/SiC composites have aroused interest for nuclear applications thanks to their high decomposition temperature, strong thermomechanical properties, neutron transparency and low swelling under irradiation. Given these properties, SiC/SiC composites are mainly considered as cladding material either for generation IV systems (such as Gas-cooled Fast Reactors) or current nuclear power plant (Light Water Reactors). Two types of fibers are currently available to manufacture nuclear grade composites: Hi-Nicalon S (HNS) and Tyranno SA3 (TSA3). Unfortunately, CVI matrix composites made of each fiber do not exhibit similar mechanical behavior. The HNS-based highlight higher ultimate tensile strains and stresses than the TSA3-based ones. Thus, both fibers have similar properties and identical interphase and matrix, which should have lead to similar behavior. The fiber/matrix coupling must have a strong influence on the final mechanical behavior. Hypotheses were made that those differences came from discrepancies in the fibers surface roughness but its decrease is not the only factor at stake. The understanding of the mechanisms controlling the fiber/matrix were then conducted in order to improve the mechanical properties of SiC/SiC composites made of TSA3 fibers. The fiber/matrix interface was characterized by a combination of mechanical testing and TEM investigations. The differences in the composition of the fiber surface were quantified by XPS and IGC. Results have pointed out that the carbonaceous structure of the fiber extreme surface has to be responsible of those mechanical behaviors.

Topic: 3 - Interfaces and interphases
Abstract no. 2069

# Rapid PyC interphase deposition on SiC fibre tows from ethanol in a cold wall reactor

Presenting author: M FENETAUD Pierre
Affiliation: LCTS
3 allée de la Boétie,33600 PESSAC (France)
Co-authors: VILLATTE Lucas (LCTS), DELEHOUZÉ Arnaud (LCTS), JACQUES Sylvain (LCTS - CNRS)

SiC/SiC ceramic matrix composites offer a new alternative to monocrystalline superalloys used for high temperature parts in aeronautic field. Those new materials gather low density, high thermal stability and resilience due to a pseudo-ductile mechanical behaviour. This specific damage mechanism is generally provided by the implementation of an interphase composed of pyrolytic carbon deposited in the fibrous preform by CVI in a batch process in hot wall reactors. This process induces an important thickness gradient within the interphase layers across the preform. A new method is proposed to elaborate pyrocarbon interphases with a constant thickness. It consists of a rapid single tow deposition process using ethanol as carbon precursor. The final purpose is to develop a continuous way to deposit a uniform interphase on a SiC fibre tow that is transported at high speed through a deposition reactor before weaving the preform. The SiC fibres are heated through of direct coupling heating process in the presence of ethanol vapours. This particular system offers a fast and localised heating while operating in a cold wall reactor, leading to high growth rate deposition on the hot tow.
Pyrocarbon thin films of several micrometers in thickness have been deposited on silicon carbide fibres with this process, for deposition times of 5 - 30 minutes at 900 – 1300 °C from ethanol under N2 flow. Scanning electron microscopy has been used to measure the film thickness while the degree of structural anisotropy of the pyrocarbon films was characterised by polarised light microscopy.

Topic: 3 - Interfaces and interphases
Abstract no. 2256

# Enhancement of Interface Interactions of SiC Fiber Reinforced Composites in Cooperation with 'Fuzzy Fiber' BNNTs coating onto SiC Fiber.


Presenting author: Mme TOP Aysemin
Affiliation: Chemical and Metallurgical Engineering of Faculty, Istanbul Technical University ITU, Ayazaga Kampüsü,Maslak,Sariyer,34437 Istanbul (Turkey)

Co-authors: KOKEN Deniz (Faculty of Engineering and Natural Science Sabanc? University Istanbul Tuzla 34956 Turkey), TURGUT F?rat (Faculty of Aeronautics and Astronautics Istanbul Technical University 34437 Istanbul Turkey), BOZALI Beyza (Faculty of Textile Technologies and Design Istanbul Technical University Istanbul 34469 Turkey), OZDEN YENIGUN Elif (5School of Design Textiles Royal College of Art SW7 2EU London United Kingdom), CEBECI Cakmak Fevzi (Faculty of Engineering and Natural Science Sabanc? University Istanbul Tuzla 34956 Turkey), CEBECI Hülya (Faculty of Aeronautics and Astronautics Istanbul Technical University 34437 Istanbul Turkey)



Stress induced cracks and oxidation is one of the most critical factor to be prevented for acquiring the potential of silicon carbide (SiC) fibers for extreme environment applications. For ceramic matric composites (CMCs), engineering the interface in between SiC fiber (SiCf)-SiC matrix will play a significant role on the final properties of overall structure. Hence, coating SiCf with an interphase layer has been adopted in literature to enhance the F/M interface interactions, prevent catastrophic fiber failure and also to protect the fibers at oxidative or any harsh environment. Pyrolytic carbon (PyC), zirconia, (hexagonal boron nitride) h-BN etc. are commonly used as the interphase materials for CMCs.
In this study, boron nitride nanotubes (BNNTs) a unique interface design for fiber reinforced CMCs were grown onto SiCf to form "fuzzy fiber" architecture that creates high temperature resistance and as well as increase surface area to provide good interface interactions. "Fuzzy fiber" BNNTs were successfully grown onto SiCf via single step vapour trapping boron oxide-chemical vapour deposition (VT-BOCVD) at 1200°C following after a simple and low-cost catalysing method. Wettability problem of BNNTs due to the apolar nature, was solved by plasma treatment which is simple method compared to chemical processes and also provides cleaner and active surface. The single fiber pull-out test results demonstrated that interface interactions were enhanced by 'fuzzy fiber' BNNTs onto SiCf. Preliminary interfacial shear strength (IFSS) calculations verified more strong F/M bond by significant increase of 25.3% and 47.3% of IFSS and load carrying capacity, respectively.

Topic: 3 - Interfaces and interphases
Abstract no. 2020

# INFLUENCE OF THE THICKNESS AND CRYSTALLINITY OF A BN INTERPHASE ON THE MECHANICAL PROPERTIES OF A MODEL CERAMIC MATRIX COMPOSITE.

Presenting author: Mme DELPOUVE Héloïse
Affiliation: Safran Ceramics
3 allée de la Boétie,33600 PESSAC (France)
Co-authors: JACQUES Sylvain (CNRS), CAMUS Gérald (CNRS), DELEHOUZE Arnaud (Safran Ceramics)

Compared with metallic materials, ceramic matrix composites (CMCs), and particularly SiCf/SiC materials, are suitable candidates for aeronautical applications with improved performance. In CMCs, the intensity of the fibre/matrix coupling depends as much on mechanical phenomena as on the physico-chemical nature of the interfacial zone. The interposition between the fibre and the matrix of an additional phase, called interphase, is a means to control the intensity of this bond. This interfacial bond is involved in all the macroscopic mechanical properties, in particular in tension and in shear. This last case, not yet studied, makes it possible to identify the initial shear modulus G°12 and its evolution with the damage. In this work, boron nitride interphases were deposited by gaseous route (CVI) in SiCf/SiC woven 2D monostrates with two microstructures (low and high degree of crystallization) and two different thicknesses (200 and 500 nm) in order to better understand their influence on the mechanical behaviour of these model CMCs. The matrix was then formed by CVI and silicon melt infiltration. A thorough characterization of the material prepared before the damage was carried out initially. Macroscopic tensile and shear tests were then adapted to the model specimens and carried out in the main axis of the reinforcing fibres. Then, push-out tests were carried out to better understand at the microscopic scale the influence of the microstructure and the thickness of the interphase on the interfacial bonds. After mechanical stress, the specimens were again characterized, in particular by scanning and transmission electron microscopy, to determine the mechanisms of ruin of the material. The four interphases tested do generate different mechanical behaviours.

Topic: 3 - Interfaces and interphases
Abstract no. 2241

# Mechanical Properties of SiCf/SiC Composites with BN Interphase Formed by Electrophoretic Deposition


Presenting author: Prof YOSHIDA Katsumi
Affiliation: Tokyo Institute of Technology
2-12-1, Ookayama, Meguro-ku,1528550 Tokyo (Japan)

Co-authors: KASAKURA Mayuko (Tokyo Institute of Technology), SHIRATA Ryo (Tokyo Institute of Technology), AJITO Takashi (Tokyo Institute of Technology), YANO Toyohiko (Tokyo Institute of Technology), KOTANI Masaki (Japan Aerospace Exploration Agency (JAXA)



Continuous silicon carbide fiber-reinforced silicon carbide matrix (SiCf/SiC) composites have been recognized as key materials for aerospace industries, high-temperature gas turbines and future nuclear and fusion applications. The fiber/matrix interfaces act as an important role for toughening and strengthening SiCf/SiC composites, and carbon or hexagonal-boron nitride has been applied as the interphase for SiCf/SiC composites. Present authors have developed novel process to form interphases on SiC fibers in SiCf/SiC composites based on electrophoretic deposition (EPD) method. In this study, the authors focused on BN interphase coating on SiC fibers by EPD method, and various types of BN particles with different particle size were used for the formation of BN interphase by EPD for SiCf/SiC composites. The effect of BN particle size on the microstructure of the BN interphase on SiC fibers and mechanical properties of the SiCf/SiC composites with BN interphase was discussed.

Topic: 3 - Interfaces and interphases
Abstract no. 2057

# Oxidation/corrosion of BN interphase in SiC/BN/SiC composites: relationship with the degree of crystallization and the orientation of crystallographic structure


Presenting author: Dr JACQUES Sylvain
Affiliation: LCTS - CNRS
3 allée de la Boétie,33600 PESSAC (France)
Co-authors: JACQUES Sylvain (LCTS - CNRS), REBILLAT Francis (LCTS - University of Bordeaux)



In the last generation of SiC/SiC composites, BN becomes the main interphase material used. Compared to carbon, it has a better oxidation resistance due to the formation of a condensed oxide phase. However, the passivation efficiency is limited in the presence of moisture. The corrosion resistance of turbostratic BN coatings processed by CVD was investigated under moist air at different temperatures using samples with two different geometries: on flat Si wafers or in a sandwich structure between two CVD SiC layers in honeycomb SiC substrates. These two geometries allowed oxidation resistance to be tested in two different main crystallographic directions: perpendicular and parallel to the sp2-BN basal planes characterized mainly by strong covalent and weak electrostatic bonds, respectively. A relationship between crystallographic organization, orientation of atomic planes and reactivity at high temperature in an oxidizing/corrosive environment is established from coatings several micrometres thick with different degrees of crystallization. If the reactivity perpendicular to the basal planes is highly dependent on the degree of crystallization, that parallel to these planes is almost not. Thus, the degree of crystallization of a BN interphase does not appear to be the key factor to improve the oxidation resistance of SiC/SiC composites. Once the thickness of the BN layer is reduced to 0.5 micrometre, typical of an interphase, in the multilayered SiC/BN/SiC architecture, the simultaneous oxidation of SiC contributes to the formation of a borosilicate sealant glassy phase, improving the protective efficiency. However, this protection is highly dependent on the oxidation temperature: effective at 800°C, it becomes limited to 1000°C. To extend the passivation domain of BN, the introduction of Al into BN is proposed. Finally, the oxidation resistance of these new B(Al)N coatings is compared to that of BN.

Topic: 3 - Interfaces and interphases
Abstract no. 1991

# Effects of Boria on Rare Earth Environmental Barrier Coatings

Presenting author: Mme GUARRIELLO Rachel
Affiliation: University of Virginia
211 Todd Avenue,22903 Charlottesville (VA, United States)
Co-authors: OPILA Elizabeth (University of Virginia)

SiC/SiC CMCs (Silicon Carbide Ceramic Matrix Composites) have recently come into use for hot section components of turbine engines. It is important to understand any reactions between the composite substrates and the Environmental Barrier Coatings (EBCs) used to protect them in these high temperature combustion environments. Currently, these CMCs are comprised of SiC fibers surrounded by a BN (boron nitride) interphase, in a SiC matrix. The BN interphase oxidizes at high temperatures to form B2O3 (boria) on the surface of the CMC. Boria is a strong glass-former when in contact with other oxides such as the ytterbium disilicate (YbDS) currently used as an EBC. Little is known about phase equilibria and reactions in the Yb2O3-SiO2- B2O3 system. One of the studies utilized for this study is an idealized interface test between the oxide constituents. Wells are drilled into Spark Plasma Sintered (SPS) or Air Plasma Sprayed (APS) samples of the YbDS coating materials, and then filled with plugs of pure boria glass. Samples are exposed in air at temperatures up to 1200C and times up to 50 hours. Scanning Electron Microscopy and Energy Dispersive Spectroscopy (SEM & EDS) are used to examine the multiple reactions zones and glass left after the exposures. The remaining glass is digested off the substrate using warm water and analyzed using Inductively Coupled Plasma - Optical Emission Spectroscopy (ICP-OES) to measure the amount of dissolved silicon and ytterbium. XRD is used to determine the phases resulting from Si and Yb depleted YbDS. The reactions and products observed will inform both phase equilibria in the Yb2O3- SiO2-B2O3 system and interactions between the EBCs and boria formed during CMC oxidation.

Topic: 3 - Interfaces and interphases
Abstract no. 2242

# Oxidation resistant Yb sislicate fiber coating system for SiC/SiC composites


Presenting author: Dr GOTO Ken
Affiliation: Institute of Space and Astronautical Science, Japan aerospace exploration agency
3-1-1, Yoshinodai, Chuo-ku,229-5210 Sagamihara (Kanagwa, Japan)
Co-authors: ITO Akihiko (Yokohama National University), MATSUDA Tetsushi (Japan Fine Ceramics Center), KITAOKA Satoshi (Japan Fine Ceramics Center)



Yb silicate (Yb2Si2O7) was examined as an interface coating for SiC fiber reinforced SiC composite to have superior anti-oxidation and water vapor. Yb2Si2O7 was selected as targeted coating with its higher resistant property for high temperature water vapor oxidation. Mechanical properties of coated fibers were examined by tensile test of a fiber bundle mini-composite. Dense Yb2Si2O7 single coating was successfully fabricated by the two coating methods, sol-gel process and CVD. However, tensile strength of a mini-composite made by sol-gel process and CVD became half of the tensile strength of a mini composite without coating. Coating was improved to possess weak bonding between fiber and dense coating by applying porous Yb silicate first layer by CVD. Finally, the properly controlled fiber coating was realized and tensile strength of mini-composite maintained original fiber strength. Finally, the coating performance was proved by a SiC/SiC bundle mini-composite fabricated by polymer infiltration and pyrolysis. Tensile strength of the SiC/SiC mini-composite was almost same as a SiC/SiC mini-composite with BN interface. Fiber/matrix interface property of SiC/SiC mini-composite examined by single fiber push-out test, also proved that double layered coating system realized proper debonding strength to posses high strength of SiC/SiC. Oxidation test of BN and Yb2Si2O7 coating also show higher oxidation resistivity of Yb silicate at higher temperature and also in high pressure water vapor environment.

Topic: 3 - Interfaces and interphases
Abstract no. 2218

# Tunable strength of Yb2Si2O7 interphase for different requirements in SiCf/SiC CMC: inspiration from SiCf/Yb2Si2O7 model composite investigation


Presenting author: M LV Xirui

Affiliation: Advanced Ceramics and Composites Division, Institute of Metal Research, Chinese Academy of Sciences
72 Wenhua Road,110016 Shenyang (Liaoning, China)

Co-authors: WANG Jingyang (Institute of Metal Research Chinese Academy of Sciences), ZHANG Jie (Institute of Metal Research Chinese Academy of Sciences), WANG Jiemin (Institute of Metal Research Chinese Academy of Sciences)



Rare earth (RE) disilicates, RE2Si2O7 are promising interphase materials in SiCf/SiC CMC for extreme combustion environment. Compared with typical PyC and BN interphases, RE2Si2O7 may endow better resistances to high temperature oxidation and hot steam corrosion. In the present work, we processed model composites consisting of SiC fiber embedded in Yb2Si2O7 matrix by Spark Plasma Sintering method and estimated the feasibility of this disilicate as interphase in SiC-based CMCs. Weak and strong SiCf/Yb2Si2O7 interfaces were designed by adopting the sintering temperatures at 1200 oC, 1250 oC, and 1450 oC. Fiber push out experiments clearly shows the different debonding mechanisms in the samples. Model composites sintered at 1200 and 1250 oC exhibit low debond energy at the interface, which are comparable to those of PyC or BN interfaces and ensure the well-recognized crack deflection criteria of energy release rate. The samples also demonstrate crack deflection at the interface in Vickers indentation test, that is also crucial for the key role of weak interface in CMCs. Strong interface with high debond energy and crack penetration into SiC fiber through the interface is disclosed in model composite sintered at 1450 oC. This work convey the information that it is possible to design the capability of RE2Si2O7 interphase for different requirements by adjusting interfacial strength or debond energy to reach optimal mechanical fuse mechanisms in SiCf/SiC CMC.

Topic: 3 - Interfaces and interphases
Abstract no. 1638

# Wet-chemical deposition of BN, SiC and Si3N4 interphases on SiC fibers

Presenting author: Dr NöTH Andreas
Affiliation: Fraunhofer Center for High Temperature Materials and Design HTL
Gottlieb-Keim Str. 62,95448 Bayreuth (Bavaria, Germany)

Co-authors: MAIER Jonathan (Fraunhofer Center for High Temperature Materials and Design HTL), KLEMM Hagen (Fraunhofer Institute for Ceramic Technologies and Systems IKTS), SCHOENFELD Katrin (Fraunhofer Institute for Ceramic Technologies and Systems IKTS)

To realize crack deflection and damage-tolerant fracture behavior in SiC/SiC CMCs, an interphase coating is typically used for appropriate bonding between the fiber and the matrix. Usually, the fiber coatings are applied by chemical vapor deposition (CVD). In this paper, we present an alternative approach to coat SiC fibers by a wet-chemical route.
First, we describe the coating technology, which is simple to upscale at low investment costs. The technology allows to coat the fibers at a speed of several hundred meters per hour applying multiple coating layers in one roll-to-roll run.
Based on this technique, we developed BN, BN/SiC and BN/Si3N4 coatings on SiC fibers (Hi Nicalon Typ S). We used the design of experiments (DoE) to optimize the deposition process varying process parameters like draw speed, precursor concentration, stabilization and pyrolysis temperature. We discuss the criteria for the evaluation of the DoE results and present details about coatings with different quality.
In order to evaluate potential fiber damage during the coating cycles, tensile tests of fiber bundles were performed. We compare the tensile strength of non-coated fiber bundles in their as-received state with fiber bundles coated with the different interphase systems mentioned above. In order to simulate the temperature load during matrix processing via the Liquid Silicon Infiltration (LSI) process, the fiber bundles were thermally annealed at 1500 °C and 1650 °C. We demonstrate that the coated fiber bundles have similar tensile strength values as their non-coated counterparts after this thermal annealing. Finally, properties of CMCs fabricated with the different fiber coatings will be presented.

Topic: 3 - Interfaces and interphases
Abstract no. 2603

# Strengthening and toughening of dense ceramic matrix composites


Presenting author: M LIU Chidong
Affiliation: Northwestern Polytechnical University
No.127 Youyi West Road,Xi'an, Shaanxi Province, China,710072 Xi'an (Shaanxi, China)
Co-authors: FAN Xiaomeng (Science and Technology on Thermostructural Composite Materials Laboratory Northwestern Polytechnical Uni- versity Xi’an), YIN Xiaowei (Science and Technology on Thermostructural Composite Materials Laboratory Northwestern Polytechnical Uni- versity Xi’an)



Dense ceramic matrix composites (CMCs) were fabricated by reactive melt infiltration (CMCs). Compared with porous matrix, the dense matrix makes the CMCs have better environmental resistance, but the higher fabrication temperatures caused the high thermal residual stress (TRS), so the strength and toughness of dense CMCs need to be improved. In order to solve the question, our group developed two solutions in the last decade. The first one is to introduce the high damage-tolerant MAX phases into matrix by the low-temperature densification process. The plastic deformable MAX phases can enrich the toughening mechanism to alleviate the TRS, so strength and toughness of dense CMCs containing MAX phases can reach to the level of porous CMCs. Another one is to increase the interphase thickness, and the TRS can be reduced by the thicker interphase, so the tensile strength of dense CMCs can reach to be 260 MPa. Through the enrichment of the matrix toughening mechanism and the optimization of interphase thickness, the strength and toughness of dense CMCs can be effectively improved, which can expand the application fields of dense CMCs.

Topic: 3 - Interfaces and interphases
Abstract no. 1910

# Fabrication of BN Interphase for SiC/SiC Composites via an Improved Dip-coating Method

Presenting author: Dr HUANJUN Song
Affiliation: Research Institute of Aerospace Special Materials and Processing Technology
Yungang Street,Fengtai District,100074 Beijing (China)

Co-authors:

SiC fiber reinforced ceramic matrix composite (SiC/SiC) is the key material to replace nickel based superalloy for the new generation aero-engine. Compared with high-temperature alloy materials, SiC/SiC composite shows outstanding properties of high temperature resistance, low density and high strength, which can significantly improve thrust-weight ratio of the aero-engine. The working temperature of SiC/SiC composite can increase by more than 400? compared with high-temperature alloy based on nickel?while the density decrease above 30 percent. Boron nitride (BN) interphase plays an crucial role in the mechanical strength and oxidation resistance of SiC/SiC composites, since the hexagonal crystal of BN interphase could help to deflect cracks from the matrix and protect the fiber from oxidation environment. Dig-coating is one of the important methods to fabricate BN interphase, for the low cost and equipments requirements of the method. In previous studies, thickness of BN interphase fabricated via dig-coating method is small, and the interphase fails to strengthen mechanical property of the SiC/SiC composite efficiently. In this work, a modified dig-coating method is introduced to fabricate high purity and large thickness BN interphase. Boric acid and urea are used as Boron precursor and Nitrogen precursor respectively. At the high temperature treatment process, a semi-enclosed container with solid urea is introduced to control the concentration of NH3 which is decomposed from urea. BN interphases with thickness from 300nm to 2000nm are fabricated in a controlled way?and XRD, SEM and EDS analysis are presented to character the composition and morphology. Additionally, the mechanical property of the SiC/SiC composites is obviously strengthened after coating the fiber with the BN interphase. By comparing the properties of composites with different thickness of BN interphase, SiC/SiC composites with BN interphase of 500nm thickness exhibit the best performance. This study will potentially be applied to preparation of high-temperature component of aeroengine.

Topic: 3 - Interfaces and interphases
Abstract no. 2453

# Formation of BN interphase for SiCf/SiC composites using flaked BN suspension by electrophoretic deposition method


Presenting author: Prof YOSHIDA Katsumi
Affiliation: Tokyo Institute of Technology
2-12-1, Ookayama, Meguro-ku,1528550 Tokyo (Japan)

Co-authors: KASAKURA Mayuko (Tokyo Institute of Technology), YOSHIDA Katsumi (Tokyo Institute of Technology), KOTANI Masaki (Japan Aerospace Exploration Agency (JAXA)



Continuous silicon carbide fiber-reinforced silicon carbide matrix (SiCf/SiC) composites have been attracting attention as next-generation heat resistant materials with high reliability used in aerospace and energy fields. SiCf/SiC composites consist of SiC fibers, SiC matrix and interphase between them. Formation of the interphase with the optimum thickness between fibers and matrix has been recognized as one of the important issues for SiCf/SiC composites to achieve excellent mechanical properties and high reliability. Generally chemical vapor deposition (CVD) or chemical vapor infiltration (CVI) method has been used as the interphase formation process for SiCf/SiC composites. Our research group focused on electrophoretic deposition (EPD) method to form hexagonal-boron nitride (h-BN) and carbon (C) interphases on SiC fibers, and we have demonstrated that EPD method is effective to form h-BN and C interphases on SiC fibers. In this study, h-BN with excellent oxidation resistance was selected as the interphase for SiCf/SiC composites and we tried to form homogeneous and dense h-BN layers on SiC fibers by EPD method using flaked h-BN suspension prepared by wet-jet milling. In addition optimum EPD conditions for h-BN coating on SiC fibers by EPD method was discussed.

Topic: 3 - Interfaces and interphases
Abstract no. 1852

# Effect of the interfaces on the mechanical properties of TiC-Ni composites fabricated by selective carburization of Ti-Ni alloys


| | |
|---|---|
| Presenting author: | Dr HANJUNG Kwon |
| Affiliation: | Korea Institute of Geoscience and Mineral resources<br>124, Gwahak-ro,Yuseong-gu,34132 Daejeon (Daejeon, South Korea) |
| Co-authors: | CHAN-YUL Suh (Korea Institute of Geoscience and Mineral resources) |



Mechanical properties of carbide/metal composites are dependent on both intrinsic properties of carbide/metal components and interfacial structure between carbide and metal phases. Thus, we should strengthen the properties of the components and interfaces to improve the properties of the carbide/metal composites. However, it is difficult to control the intrinsic properties of the components in the composites because the intrinsic properties of carbide and metal phases are fixed. Also, the fracture of composites is mainly caused by cracking in the interfaces between carbide and metal phases. Therefore, the best way to enhance the mechanical properties of composites is strengthening of the interfaces. In this study, carbide/metal composites with strong interfacial structure between carbide and metal phases were fabricated by selective carburization of titanium alloys. The improved properties of composites prepared by the selective carburization was proved by observation of interfacial structure and evaluation of fracture toughness of the composites.

Topic: 3 - Interfaces and interphases
Abstract no. 2002

# Tailoring the Fiber/Matrix interface in Continuous Alumina fiber reinforced Zirconia composites for Mechanical Properties


Presenting author: Prof NI Na
Affiliation: Shanghai Jiao Tong University
800 Dongchuan Road,200020 Shanghai (Shanghai, China)
Co-authors: LI Xuewu (Shanghai Jiao Tong University), FAN Xiaohui (Shanghai Jiaotong University)



Conventional continuous oxide fiber reinforced ceramic matrix composites such as aluminaf/alumina or aluminaf/mullite typically employ porous matrix in order to achieve an adequate fracture toughness of the composite. A tougher matrix, such as zirconia, may reduce the need for a porous matrix and thereby increase the resistance of the material to environmental degradation such as oxidation. Alumina fiber reinforced zirconia composites could offer promising high temperature mechanical properties due to the high elastic modulus (370–380 GPa) and tensile strength (2.5–3.3 GPa) of alumina fiber combined with the good fracture toughness of zirconia. In this work, continuous alumina fiber-reinforced yttria-stabilized zirconia (Al2O3,f/YSZ) composites were fabricated by slurry infiltration and spark plasma sintering (SPS) process. The focus was to investigate the mechanical properties as a function of the stress state at the fiber/matrix interface, which is tailored by heat treatments and the application of a novel reduced graphene oxide (rGO) coating on the fiber. When no coating was applied, it was revealed that heat treatments at different temperatures (1200-1400) for different time (5-10 min) resulted in a large variation of the stress at the fiber/matrix interface, which cannot be explained solely by the thermal mismatch of the materials. The rGO coating can be successfully prepared and changes the interface stress state dramatically. The fracture mode and mechanical properties of the composites are investigated and discussed in light of the interface structure and stress state. The results show that when a proper design of the fiber/matrix interface is achieved, Al2O3,f/YSZ composites can offer a good fracture toughness with the potential to be more oxidation resistant due to the use of a dense matrix.

Topic: 3 - Interfaces and interphases
Abstract no. 2418

# BN CVD for continuous ceramic fiber


Presenting author: M KAWAHARA Fumitomo
Affiliation: ADMAP INC.
Tamahara 3-16-2,706-0014 Tamano (Okayama, Japan)
Co-authors: SHIBATA Kenichi (ADMAP Inc.)



ADMAP Inc. has been developing h-BN CVD on ceramic fiber. And also we are investigating the possibility of the h-BN / SiC coating on ceramic fiber.
We will report some investigation results of these developments.

Topic: 3 - Interfaces and interphases
Abstract no. 2111

# Preparation of SiC/SiC minicomposite with ZrO2 interface and evaluation of interfacial mechanical properties


Presenting author: M IKEDA Kenyu
Affiliation: Tokyo University of Science
Hatanodai?2-10-14,142-0064 Shinagawa City (Tokyo, Japan)

Co-authors: GOTO Ken (JAXA), KOGO Yasuo (Tokyo University of Science), INOUE Ryo (Tokyo University of Science), ITO Akihoko (Yokohama National University)



In this study, SiC/SiC composites having ZrO2 interface was prepared and the interfacial mechanical properties were evaluated. Composite consisting of SiC fiber bundle (Tyrano SA: 1600, Ubekyosan) was prepared for SiC/SiC. From Zirconium(IV) Acetylacetonate, ZrO2 was prepared on the fiber surface by laser CVD method. ZrO2 was prepared at various temperatures by changing laser output. Two ZrO2 interfaces having different fabrication temperatures were also prepared. SiC/SiC minicomposites were formed by Polymer Impregnation and Pyrolysis (PIP) method. The fiber bundle was impregnated with a precursor of SiC (Polycarbosilane, Starfire) and then heat treatment at about 1,000 °C, for 1 hour under an Ar atmosphere. In order to directly measure the function of ZrO2 as the interface a single fiber Push-out test was carried out. The resin-filled SiC/SiC was polished to a thickness of about 100 ?m. Interfacial shear strength was calculated by pushing out single fibers with a Vickers indenter. Further, a Push-in test was carried out on a specimen polished to about 1mm to obtain the interfacial debonding energy.

# Topic: 4

# Innovative Design, Advanced Processing, and Manufacturing Technologies processing in composites: Oxides & Geopolymers (incl. short fibers)

Topic: 4 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in composites : Oxides & Geopolymers (incl. short fibers)
Abstract no. 2038

# All-oxide ceramic matrix composites (OCMC) based on low cost 3M™ Nextel™ 610 fabrics


Presenting author: M PRITZKOW Walter

Affiliation: Walter Pritzkow Spezialkeramik
Adam Opel Str. 6,70794 Filderstadt (Germany)

Co-authors: VISSER Larry (3M), D'SOUZA Andrew (3M), REZWAN Kurosch (Advanced Ceramics University of Bremen), TUSHTEV Kamen (Advanced Ceramics University of Bremen)



Searching for possibilities to reduce the costs of Nextel™ 610 fabrics, the company 3M™ designed a large number of new experimental fabrics. The aim of the present study is to investigate alternatives to the conventional Nextel™ 610 fabric DF11 giving the same or better characteristics in OCMC. A cost reduction can be reached using higher denier roving but getting the same fabric areal weight and the same fabric thickness. Fabrics based on 3000 denier and 4500 denier with different weavings were used to produce OCMC with alumina-zirconia-matrix. Allso semi-uni fabrics based on 10.000 denier and 20.000 denier in warp and a 1500 denier roving in fill were used for the OCMC production.

The study covers a number of aspects of the new OCMC including the preparation of composites, their microstructure and mechanical performance. In plane and interlaminar properties of the composites were evaluated under tensile, compression, and flexural load. Furthermore, the fracture toughness and the notch sensitivity were determined by on-edge flexural and SENB tests. The experiments indicate that the application of higher-denier fabrics with 0/90° architecture leads to composites with mechanical properties comparable to those of the commertial OCMC FW12. Considerably higher composite strength was achieved using semi-uni fabrics. The resultant composite properties are discussed in consideration of fiber denier and fabric architecture.

Topic: 4 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in composites : Oxides & Geopolymers (incl. short fibers)
Abstract no. 2048

# Development of Ox/Ox composites for LPT components - Effect of abradable TBC


Presenting author: M SEKIGAWA Takahiro

Affiliation: Mitsubishi Heavy Industries Aero Engines
1200, Higashi-Tanaka,485-0826 Komaki city (Aichi Pref., Japan)

Co-authors: FUKUSHIMA Akira (Mitsubishi Heavy Industries Aero Engines), NOGAMI Ryoma (Mitsubishi Heavy Industries Aero Engines), TAMUGI Azusa (Mitsubishi Heavy Industries Aero Engines), NISHIKAWA Kosuke (Mitsubishi Heavy Industries), KURIMURA Takayuki (Mitsubishi Heavy Industries), MATSUMOTO MIneaki (Mitsubishi Heavy Industries), KAKISAWA Hideki (National Institute for Materials Science), HASEGAWA Yoshio (Art Kagaku Co.), KUMETA Kazuhiro (Nitivy Co.)



Future gas turbine engines are aimed for higher efficiency with lower exhaust emissions. Efficiency of the gas turbine engine increases with an increase in turbine inlet temperature (TIT). Current Nickel based super-alloys are operating at their maximum temperatures and cannot withstand an increase in temperature. Ceramic matrix composites (CMCs) are one of the best candidate materials because of their low weight and high temperature capability. In order to successfully implement CMCs in gas turbine engines, the cost affordable and environmental superior Ox/Ox composites have been developing. This paper focuses on improvement of abradable thermal barrier coatings (TBC) having the compatibility with CMCs and the test results of exposure by laser ablation to heating and cooling of 1,000 cycles at temperatures similar to those of gas turbine.


This research was supported by the Structural Materials for Innovation of the Cross ministerial Strategic Innovation Promotion Program (SIP) of Japan Science and Technology (JST).

Topic: 4 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in composites : Oxides & Geopolymers (incl. short fibers)
Abstract no. 2204

# New Developments in Oxide-Oxide Ceramic Matrix Composites Manufacturing

Presenting author: M JACKSON Barrett
Affiliation: Composites Horizons
135 NE 45th Ave,97213 Portland (OR, United States)
Co-authors: SIMPSON Marc (3M)

New Developments in Oxide-Oxide Ceramic Matrix Composites Manufacturing

B. Jackson
Composites Horizons Inc.
Covina, CA
M. Simpson
3M Company
St. Paul, MN

This is very exciting time to be working with Oxide-Oxide Ceramic Matrix Composites. After years of developing material properties, fabricating test components and the beginning of some commercial production the interest in this class of materials is growing rapidly. To further the development of Oxide CMC's for continued growth, 3M the maker of 3M™ Nextel™ Ceramic Fibers and Composites Horizons Inc., CMC parts designer and fabricator, have partnered to further the development of new higher denier (3000 denier and 4500 denier) fabric weaves. These new weaves, utilizing higher denier tow, show a reduction in material and fabrication costs, as compared to the standard 1500 denier 8HS weave. This paper will present the results of current material testing and an evaluation of manufacturing highly complex components with higher denier fabrics. Emphasis will be given to 4500 denier weaves, especially 2x2 twill.

Topic: 4 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in composites : Oxides & Geopolymers (incl. short fibers)
Abstract no. 2166

# 2D-Laminated Oxide-Oxide FRCMC with Tailored Internal and Surface Structure via Polmyer-based Prepregs


Presenting author: Dr JANSSEN Rolf
Affiliation: Institute of Advanced Ceramics, TU Hamburg
Denickestr. 15,21073 Hamburg (Germany)

Co-authors: BLAESE Diego (Institute of Advanced Ceramics Tu Hamburg), GUGLIEMLI Paula (Helmholtz Center geesthacht)



Precise microstructural tailoring is the most essential prerequisite for aadvanced ceramic composites offering advanced mechanical performance and reliability at elevated temperatures. This holds particularly for components made out of composites with complex hierarchical structure, e.g. fiber reinforced ceramics (FRCMC). In the past, we´ve developed at TUHH a new processing route for FRCMC based on the lamination of thermoplastic prepregs which allows not only affordable manufacturing but also precise tailoring of the internal structure (both intra-bundle and inter-textile) as well as the surface topography of complex shaped products.

Basically, the route integrates processing techniques well known in the ceramic community: (a) infiltration of fibre-bundles and -textiles with low viscous suspensions, (b) manufacturing of 2 D prepregs using commercial fibre fabrics impregnated with compounds of ceramic particles embedded in an organic matrix, (c) followed by respective stacking, (d) burn out of the organic matrix, (e) preconditioning for subsequent coatings, (f) dip-coating of the compound with highly doped and therefore sinter-active suspensions and (g) final heat treatment to sinter both the FRCMC as well as the coating in one step.

In the presentation, the focus is on $Al_2O_3/ZrO_2$ matrix reinforced by NEXTEL 610 textile, denier 1500 using the porous matrix approach to achieve the desired interface crack deflection. Details of the processing steps will be outlined and Composites of 8 layers withstand bending stresses > 400 MPa followed by a graceful failure behavior, e.g. a stepwise stress reduction after peak nominal stress caused by a series of interfacial delamination events: The results are reasoned considering intra-bundle and inter-textile crack deflection phenomena. Thereby, generalized concepts for tailoring affordable and reliable FRCMC will be derived. Lastly, emphasis will be given to the synthesis of dense coatings enabling composite service in harsh abrasive and corrosive environments.

Topic: 4 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in composites : Oxides & Geopolymers (incl. short fibers)
Abstract no. 1913

# Ionotropic gelation as an alternative for the production and joining of oxide ceramic composites

Presenting author: Dr ALMEIDA Renato S. M.
Affiliation: University of Bremen
Am Biologischen Garten 2,28359 Bremen (Bremen, Germany)

Co-authors: FARHANDI Hedieh (University of Bremen), TUSHTEV Kamen (University of Bremen), REZWAN Kurosch (University of Bremen)

The production of complex-shaped all-oxide ceramic matrix composites (Ox-CMC) is somewhat restricted by their current processing techniques. A critical step during the processing is related to the fiber infiltration. Typically, a slurry with low particle content is used to obtain a good infiltration. However, this may lead to matrix cracking during the sintering step. To overcome these problems, we present here a new method for producing Ox-CMCs based on the gelation of slurries with the polysaccharide alginate. Ionotropic gelation presents several advantages over classic slurry infiltration methods, such as: the control of the slurry viscosity due to the gelling reaction, a relatively high ceramic particle content of the slurry, a low content of binder, as well as the possibility of joining and of using flexible shaping methods. For this investigation, Nextel 610/alumina-zirconia composites were produced using alginate as the binder and aluminum acetate as the gelling agent. The produced composite showed mechanical properties (strength and fracture toughness) in the same range as commercial Ox-CMCs. In addition, the joining capabilities of this technique were also investigated. For that, a slurry containing alginate was used to join two composite plates at different stages of the processing and the results, in terms of shear strength, were compared.

Topic: 4 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in composites : Oxides & Geopolymers (incl. short fibers)
Abstract no. 1925

# The effect of matrix porosity on the properties of Oxide Fiber Composites (OFC)


Presenting author: M PUCHAS Georg
Affiliation: University of Bayreuth - Department of Ceramic Materials Engineering
Ludwig-Thoma-Str. 36b,95447 Bayreuth (Germany)

Co-authors: WAGNER Lukas (Hochschule Hof), GORYWODA Marek (Hochschule Hof), KRENKEL Walter (University of Bayreuth - Department of Ceramic Materials Engineering)



The porosity of weak matrix composites (WMCs) and in particular of OFCs determines their mechanical properties, such as tensile and bending strength, interlaminar shear strength, compression strength, Young's modulus and, fracture toughness. While a higher porosity will lead to a higher tensile strength and a higher fracture toughness, the interlaminar shear strength and Young's modulus will decrease with increasing porosity. Therefore, by increasing or decreasing the porosity of OFC and by changing the pore size distribution, a wide range of different material properties are feasible and the composites can be tailored for different applications.

Starting with Nextel 610/$Al_2O_3$-$ZrO_2$ composites with the porosity in the range of 27-30 %, various methods were used to vary the porosity of the OFCs in the range from 20 to 45 %. The effect of porosity on the interlaminar shear strength, the bending strength and, Young's modulus was examined and the resulting microstructures were investigated. The pore size distribution and the through-thickness permeability of the composites were measured in order to determine the effect of porosity as well as pore size distribution on the permeability of selected samples.

Topic: 4 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in composites : Oxides & Geopolymers (incl. short fibers)
Abstract no. 2296

# In situ observation of elevated temperature deformation and fracture of an oxide (alumina-alumina) ceramic matrix composite


Presenting author: M PIRZADA Talha
Affiliation: University of Oxford
5 OC tanner Linacre College,Oxford,OX1 3JA Oxford (Oxfordshire, United Kingdom)
Co-authors: LIU Dong (University of Bristol), MARROW James (University of Oxford), RICHIE Robert (University of California Berkele)



Oxide ceramic matrix composites (CMCs) are used as structural materials in high temperature oxidising environments, such as gas turbine sealing applications, which require spring retention at elevated temperature. The material investigated was an oxide ceramic matrix composite, fabricated by Composite Horizons (PPC structural) with an eight harness-satin weave in the 0/90° orientation. The matrix was porous alumina (Al2O3) whereas the Nextel 720 fibres were alumina/mullite with a diameter of 10-12 µm. In situ high resolution X-ray tomography of flexural (3 point bending) tests was conducted at room temperature and at 1100°C at the Advance Light Source (ALS) at Berkeley, California, USA to study the effects of temperature on fracture behaviour. The three-dimensional displacement field within the specimens was quantified by digital volume correlation. This allowed the opening modes of the initiated cracks to be measured (modes I and II). The curvature of the loaded specimens was also measured, which allowed assessment of the effects of crack development on location of the neutral axis and the effective modulus of the specimen.

Topic: 4 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in composites : Oxides & Geopolymers (incl. short fibers)
Abstract no. 2003

# Design and Development of Complex and Large Ox-Ox CMC Components


Presenting author: Dr RUBIO Virtudes

Affiliation: National Composites Centre
Bristol Bath Science Park,Feynman Way Central,BS16 7FS Bristol (United Kingdom)

Co-authors: HEALEY Adam (National Composites Centre), DAVIES Hannah (National Composites Centre), D'ANGIO Andrea (National Composites Centre andrea.d'angio@nccuk.com), HOCKING Matt (National Composites Centre), HELENON Fabrice (National Composites Centre), STAMATOPOULOS Georgios (National Composites Centre), OSMIANI Camila (National Composites Centre)



Improved fuel efficiency through lightweight materials is a driving force behind development of materials for civil aviation engines. Fibre Reinforced Ceramic Matrix Composites (FRCMCs) combine comparatively low density with higher fracture toughness than monolithic ceramics whilst maintaining their high temperature capabilities (in excess of 1000°C). When low thermal conductivity for heat shields, aft cowls and liners is required, Oxide-Oxide CMCs (Ox-Ox CMCs) represent an optimal solution. Furthermore, their resistance to corrosive and oxidative environments make them excellent candidates to replace metallic components in gas turbine applications. Ox-OX materials also provide a favourable alternative to SiC/SiC composites due to the lower cost of manufacture.

The National Composites Centre (NCC) based in Bristol, UK, has been developing manufacturing processes for Ox-Ox CMCs to fabricate large, complex components using alumina fibres (Nextel 610) pre-impregnated with an alumina slurry matrix. A parametric analysis has been carried out on the key process variables (KPV's) associated with the curing and sintering processes in parallel with drapability and forming experiments, utilising the NCC's state-of-the-art capabilities.

Topic: 4 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in composites : Oxides & Geopolymers (incl. short fibers)
Abstract no. 1934

# High temperature mechanical behavior of an alumina/alumina composite


Presenting author: M DEBARRE Antoine
Affiliation: ONERA
29 avenue de la Division Leclerc,92320 CHATILLON (France)

Co-authors: JULIAN-JANKOWIAK Aurélie (DMAS ONERA), BOUSSUGE Michel (MINES ParisTech Centre des Matériaux), PARLIER Michel (DMAS ONERA), PUJOL Guillaume (DGA Techniques aéronautiques Matériaux et Technologies Plates-formes et Propulsion)



An increase in service temperatures is required to improve the thrust-to-weight ratio for high performance aero-engines. Thus, ceramics matrix composites materials such as oxide/oxide composites are currently developed as potential candidates for applications between 800°C and 1000°C in exhaust systems.

ONERA is developing such materials since a few years, paying a particular attention to the relationships between the fibers, the microstructure and the composition of the matrix, and the final mechanical properties. A study of the room temperature behavior has already been conducted, demonstrating that interesting strength (~270 MPa) and elastic modulus (~135 GPa) can be reached with such materials. The interest now focuses on the characterization of the mechanical behavior of this composite and on the understanding of time and environment influence on its properties at high temperature. Therefore, the relationships between the fibers, the microstructure and the evolution of the mechanical properties were investigated.

Assessment of the mechanical behavior of the material at high temperatures, through 4-point bending tests performed with constant loads and different heating rates, allowed critical temperatures and detrimental degradation mechanisms (such as sintering of the matrix, creep of the fibers, interfaces degradation…) to be determined. Meanwhile, the influence of hygrometry on the microstructure and fracture behavior was studied, in order to decorrelate the influences of temperature and of the atmosphere.

Topic: 4 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in composites : Oxides & Geopolymers (incl. short fibers)
Abstract no. 1938

# Understanding the Slurry Infiltrationand RetentionCharacteristicsofHigher DenierNextel™Fabrics in Oxide-Oxide CMC Systems


Presenting author: Dr WADE James
Affiliation: University of Birmingham
Metallurgy & Materials,University of Birmingham, Elms Road,B15 2SE Birmingham (N/A, United Kingdom)
Co-authors: EDMONDS Ian. M (Rolls-Royce PLC), BINNER Jon (University of Birmingham)



Porous oxide matrix ceramic composite systems reinforced with continuous oxide fibres (POx-CMC) offer a number of benefits over other systems. As well as providing the crack deflection and fibre pull-out mechanisms characteristic of CMCs, the materials are of lower density (containing ~30% porosity) and oxidation resistant. Moreover, these materials can be readily produced using cost-effective approaches to CMC production, namely slurry infiltration.
The aim of slurry infiltration is to homogeneously distribute powder particles throughout a network of fibres and at inter-laminar locations, eliminating undesirable microscale intra-tow porosity as well as inter-ply macro-scale porosity that can negatively affect mechanical performance. Such a process is usually controlled by the careful and considered optimisation of slurry rheology in order to control slurry flow during ceramic tow infiltration and CMC consolidation. However, in recent years, an increasing desire to exploit POx-CMCs in a wider range of applications has driven increasing interest in higher denier fabrics due to the potential cost savings. Despite the developing interest in these newer fabrics, further work is needed to understand the effect fabric denier has on the infiltration characteristics of slurries and the potential challenges they may present.

In this presentation, we describe the results of a study assessing the slurry infiltration and retention characteristics of 3MTM NextelTM 610 fabrics with increasing deniers. In assessing the efficacy of slurry infiltration and retention, conclusions are presented regarding the dynamics of slurry flow during slurry infiltration and consolidation.

Topic: 4 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in composites : Oxides & Geopolymers (incl. short fibers)
Abstract no. 2059

# Progress in industrial manufacturing of oxide fiber composites


Presenting author: Dr WAMSER Thomas
Affiliation: Schunk Kohlenstofftechnik GmbH
Rodheimer Straße 59,35452 Heuchelheim (Germany)

Co-authors: ZULAUF Lukas (Schunk Kohlenstofftechnik GmbH), LAUER Andreas (Schunk Kohlenstofftechnik GmbH)



In recent years there is an increasing interest on oxide fiber composites (Ox/Ox) because of their excellent thermomechanical behavior and corrosion resistance. However, application of these materials is often limited due to high fiber costs and reproducibility. To overcome these problems high-denier fibers were tested and a prepreg technology was developed in cooperation with universities and institutes. Therefore water-based prepregs reinforced with high denier fabrics (Nextel™610, 20.000 den) were used to produce oxide fiber composites with autoclave technology. The presentation will show that microstructure of the composites is homogeneous and fiber tows are well-infiltrated. Some mechanical characteristics are compared with specifications of composites reinforced with other fabric types. Finally, examples for application of Ox/Ox components for heat treatment of metals are given.

Topic: 4 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in composites : Oxides & Geopolymers (incl. short fibers)
Abstract no. 2292

# Evaluation of Surface Finish technology in the part manufacturing of Oxide-Oxide Ceramic Matrix Composites Parts

Presenting author: M TONTISAKIS Antonios
Affiliation: Axiom Materials
2320 PULLMAN STR,92705 SANTA ANA (CA, United States)

Co-authors: DHAWAN RAJ (AXIOM MATERIALS), LINCOLN JOHNNY (AXIOM MATERIALS)

With major development programs underway and a growing interest more complex oxide-oxide ceramic matrix composite fabric weaves and resin systems, an investment in improving the surface finish quality of the composite parts is required to satisfy the current applications.
Axiom Materials, Inc. has invented a new surfacing material which will co-cure and sinter with composite part manufactured by using Ox-Ox pre impregnated fabrics or wet-layup or infusion process.
This new surfacing material eliminates additional manufacturing processing, including curing and sintering to minimize surface porosity, smoothness, airflow and thermal cycling by minimizing microcracking. It is based on Ox-Ox chemistry compatible with the current systems in the market and able to withstand temperatures up to 1400°C.

Topic: 4 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in composites : Oxides & Geopolymers (incl. short fibers)
Abstract no. 2275

# Two examples of the usage of oxide fibres in various matrices

Presenting author: M NIKONOVICH Maxim
Affiliation: Institute of Solid State Physics of Russian Academy of Sciences
Academic's Osipyana Street, 2,142432 Chernogolovka (Moscow Region, Russia)

Co-authors: GALYSHEV Sergei (Institute of Solid State Physics of Russian Academy of Sciences), KOLCHIN Andrei (Institute of Solid State Physics of Russian Academy of Sciences), PROKOPENKO Vyacheslav (Institute of Solid State Physics of Russian Academy of Sciences), MILEIKO Sergei (Institute of Solid State Physics of Russian Academy of Sciences)

Oxide fibers used in this work were produced by the internal crystallization method (ICM). The first example of their usage in CMCs is a composite of a hierarchical macrostructure, which contains reinforcing elements of the oxide-molybdenum composite and the matrix is the same as that of the fibres. In particularly, the oxide material is Al2O3-Al5Y3O12 eutectic. Preliminary experiments have shown a possibility of creating sufficiently crack resistant structures of this type with critical stress intense factor reaching 25 MPa?m1/2.
The second example is fibrous oxide composites without the matrix. In this case, there we used sapphire fibers, obtained by ICM. The fibers have two plane side surfaces. Contacting couples of the fibers on such surfaces provides "weak" interfaces that can arrest macrocracks. Composite specimens were made by sintering package of the fibers under pressure. Preliminary test results indicate the effectiveness of such composites.

Topic: 4 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in composites : Oxides & Geopolymers (incl. short fibers)
Abstract no. 1859

# COMPOSITES SYNTHESIZED WITH ACID-BASED GEOPOLYMERS


Presenting author: Mme MATHIVET Virginie
Affiliation: ONERA
29 Avenue de la Division Leclerc,92320 Châtillon (France)

Co-authors: JOUIN Jenny (IRCER), ROSSIGNOL Sylvie (IRCER), RITTI Marie-Hélène (ONERA), PARLIER Michel (ONERA)



To reduce the cost of oxide ceramic matrix composites, the oxide matrix could be replaced by a geopolymer matrix that is cheaper thanks to the low cost of their raw materials (clays, strong acid or base). However, to avoid the fiber degradation by alkali cations, these geopolymer matrices will be synthetized in acidic medium. This new way of synthesis seems promising with improved mechanical properties and the formation of refractory phases as AlPO4 with increasing temperature.
This study focuses on the selection of the appropriate formulations of acid-based geopolymer matrices to develop composites with the best mechanical properties at room temperature and after thermal treatments. The formulation was fit to the different fiber preforms (alumina, basalt preforms). The studied matrices were synthesized with various water contents and Al/P ratios. The impact of the matrix formulation on the composites' microstructure was studied by Scanning Electron Microscopy combined with Energy Dispersive X-ray Spectroscopy. The mechanical properties of these composites were then characterized by short span bend test to determine their InterLaminar Shear Strength and by standard traction tests at room temperature and after thermal treatments.
For alumina preforms, the best mechanical properties at room temperature are obtained with matrices having low water content. However, these optimal matrices used for alumina preforms could not be directly transferred to basalt ones. Indeed, due to the higher water demand of basalt preforms, formulations with higher water contents have to be used. The impact on the mechanical properties of these various matrix formulations will be then studied according to the fiber preform and the temperature of thermal treatment.

Topic: 4 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in composites : Oxides & Geopolymers (incl. short fibers)
Abstract no. 2063

# Three innovative ways to get an oxyde oxyde composite of barium aluminosilicate as a matrix and alumina fibers as reinforcement.

Presenting author: Dr ALLEMAND ALEXANDRE
Affiliation: CEA
3 Allée de la Boetie,33600 PESSAC (France)

Co-authors: LE PETITCORPS YANN (UNIVERSITE DE BORDEAUX-LCTS), MAILLE LAURENCE (UNIVERSITE DE BORDEAUX), BESNARD CLEMENCE (CNRS-LCTS), COUILLAUD SAMUEL (GALTENCO SOLUTIONS), LEON JEAN-FRANCOIS (GALTENCO SOLUTIONS)

During Spark Plasma Sintering (SPS) of barium aluminosilicate $BaAl_2Si_2O_8$ (BAS) a major problem appeared as soon as big parts or complex shapes tried to be made. Indeed, whether it is for the sintering of pins either discs of 60 mm diameter all the tests led by SPS bring to a total or partial fusion of the sample. This fusion occurred for temperatures of instruction much lower than the melting point of the material. Tests led on samples 10 mm in diameter allowed to estimate a difference of 200°C between the temperature measured on the surface of the matrix and the real temperature of the sample. It is this impossibility to master exactly the thermal gradient during the SPS process that has brought us to work out on an ultra fast new method of sintering ceramics without pressure. This method, similar to SPS in terms of speed of temperature rise and of temperature dwell, allows a perfect control of the thermal gradient in the sample to sinter and this whatever is its geometry. This method bases itself on the manufacturing of a specific mold contains a number of heating elements piloted independently in real time to master the thermal gradient. This presentation shows a comparative study between the SPS sintering route and this new GALTENCO method to sinter BAS composite with short alumina fiber. In addition, an exploratory process of film boiling chemical vapor infiltration (FBCVI ) is under way to get small cylindrical BAS tube . In that case long alumina fibers are used as reinforcement.

Topic: 4 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in composites : Oxides & Geopolymers (incl. short fibers)
Abstract no. 2011

# Title to be completed


Presenting author: Mme BILLER Nancy-Jane
Affiliation: Hochschule Reutlingen
Alteburgstr. 150,72762 Reutlingen (Baden-Württemberg, Germany)

Co-authors: PRITZKOW Walter E.C. (Keramikblech)



Oxide Ceramic-matrix-composites (OCMC) preferred for high temperature applications due to their mechanical behaviour like bending strength, thermal shock-, corrosions- and temperature resistance are commonly reinforced with wovens or braidings. These textiles are expensive and time-consuming in manufactoring. Their high properties, especially the bending strength, are not always necessary concerning the applications, e.g. inhousing of high temperature cameras, furnace insulation or product carriers.

Within a research project in cooperation with a OCMC manufacturer funded by the German gouvernment the purpose was to develop a ceramic short fibre wet-laid nonwoven for ceramic slurry infiltration. The nonwoven should be built up with 3M Nextel 610 Al2O3 fibres in a range of 6-25 mm length.

Research design:

- fibre analysis (geometrical, surface potential)
- manufactoring and analysis of first wet-laid nonwovens
- preparing components for pretests
- preparing components for applications
- optimisation nonwoven and manufactoring process

Composites made of short ceramic fibres can be gentle and easily processed by the wet-laid technology. Other results are chopped fibres without sticky ends and excellent single fibre dispersion. Tested samples achieved 1/6 of woven reinforced bending strength.

The latest development´s mechanical behaviour for low-middle bending strength applications is suitable. If higher bending strength is required the nonwovens can be reinforced with rovings or wovens. First trials with nonwovens reinforced with rovings shows higher bending strength at lower fibre volume content as commonly reinforced OCMC´s.

The target of achieving a short fibre reinforced OCMC is obtained, they show adequate properties for low-middle bending strength applications at reduced costs up to 30-40 %.

Topic: 4 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in composites : Oxides & Geopolymers (incl. short fibers)
Abstract no. 1911

# Improving the mechanical properties of titanium carbonitride reinforced alumina ceramics by spark plasma sintering

Presenting author: Prof SZUTKOWSKA MAGDALENA
Affiliation: INSTITUTE OF ADVANCED MANUFACTURING TECHNOLOGY
WROCLAWSKA,30-011 KRAKOW (MALOPOLSKA, Poland)

Co-authors: PODSIADLO Marcin (Institute of Advanced Manufacturing Technology), CYGAN Slawomir (Institute of Advanced Manufacturing Technology), FIGIEL Pawel (Institute of Advanced Manufacturing Technology), CYBORON Jolanta (Institute of Advanced Manufacturing Technology)

Recent advances in tool materials have focused on reinforcing alumina with different carbides, oxides and nitrides in order to improve hardness, fracture toughness and wear resistance. The present study reports results obtained by reinforcing alumina ceramics with Ti(C0.3N0.7), Ti(C0.5N0.5), in amount 30 wt.% with the addition of 2wt.% ZrO2(m) prepared on the basis micro and nanoscale trade powders. A new type of sintering technique, the spark plasma sintering (SPS) method within a temperature range from 1600?C to 1700?C was used. Vickers hardness, Young modulus, apparent density, wear resistance, indentation fracture toughness KIC(HV) and fracture toughness assessed by means of the conventional method (3PB) on SENB specimens were evaluated at room temperature. An increase of the sintering temperature resulted in a decrease of hardness and an increase of the fracture toughness tested composites. Composites with addition of Ti(C0.5N0.5) phase in nanoscale reveal values of the Vickers hardness (19.5 GPa) and fracture toughness (5.0 MP? m1/2) comparable to those based on Ti(C0.5N0.5) phase in microscale. Composites with addition of Ti(C0.3N0.7) phase have lower Vickers hardness values of about 10 %. The samples were subjected to tribological tests using the ball-on-disc method. The friction coefficient was in a range from 0.20 to 0.55 depending on the sintering temperature. An increase in the sintering temperature resulted in an enhancement of the specific wear rate. Samples with the same chemical composition were also sintered with pressureless (PS) method. Despite the use of a higher sintering temperature exceeding 1700?C, mechanical properties of samples are lower than samples sintered by SPS method. X-ray diffraction patterns were presented in order to determine the phase composition. An observation of the microstructure was carried out using scanning electron microscopy.

Topic: 4 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in composites : Oxides & Geopolymers (incl. short fibers)
Abstract no. 1937

# Desing of an oxide/oxide ceramic matrix composite microstructure through controlled layup of a pre-impregnated ceramic


| | |
|---|---|
| Presenting author: | Mme ECHEVERRIA Itsaso |
| Affiliation: | National Composites Centre<br>Feyman Road,BS16 7FS Bristol (United Kingdom) |
| Co-authors: | FISHPOOL David (National Composites Centre), RUBIO Virtudes (National Composites Centre), YEOMANS Julie (University of Surrey), DOREY Robert (University of Surrey) |



Ceramic Matrix Composites (CMCs) are used for high temperature applications, such as thermal barriers or components in high efficiency engines, due to their refractoriness and sustained mechanical properties at elevated temperatures.

This work focuses on the development of a manufacturing route for a weak-matrix oxide/oxide CMC, based on established polymer matrix composite processing techniques. The principal aim of the research work is to reduce the fabrication cost. A pre-impregnated material, comprising alumina fibres and alumina matrix was used. The material was laid-up by hand and three different consolidation techniques were investigated: vacuum bagging with oven drying, warm pressing and autoclave processing. The resulting green bodies were then sintered at temperatures between 1100°C - 1200°C for a range of sintering times. The results of 4-point bending test showed strong dependency on the manufacturing process and specifically the level of macro porosity in the laminate. It was demonstrated that decreasing levels of macro-porosity resulted in increased flexural strength, with the highest strength being three times greater than the lowest. Autoclave processing resulted in the lowest macro-porosity and the most homogeneous pore spatial distribution, as measured by X-ray computed tomography, and correspondingly produced the specimens with the highest flexural strength. Nevertheless, despite the low macro-porosity, the strength recorded was slightly below the average values reported in literature for equivalent material systems. Further work is on-going to improve and eventually optimise the CMC processing route.

Topic: 4 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in composites : Oxides & Geopolymers (incl. short fibers)
Abstract no. 2180

# Design, manufacture and testing of an alloxide fiber reinforced fan blade


Presenting author: Prof KOCH Dietmar

Affiliation: German Aerospace Center, Institute of Structures and Design
Pfaffenwaldring 38-40,70569 Stuttgart (Germany)

Co-authors: SHI Yuan (German Aerospace Center Institute of Structures and Design), PRITZKOW Walter (Walter E. C. Pritzkow Spezialkeramik), HOLZDEPPE Dieter (TLT-Turbo GmbH), JANSSEN Rolf (Hamburg University of Technology Institute of Advanced Ceramics)



Alloxide fiber reinforced composites are well known for their damage tolerant behavior up to temperatures of 1000°C. These properties make them attractive for various industrial applications, not only under extreme conditions but also at moderate temperatures when the overall properties are beneficial. One example is a hot gas rotor which will be used in rough industrial processes especially with gas temperature of also far above 600 °C and which must withstand temperature and erosion under long term conditions. In this framework a fan blade prototype was designed, manufactured, tested and evaluated via finite element analysis. First, prepregs are developed from oxide ceramic matrices and oxide fibers. These prepregs enable serial production as they can be produced in a reproducible quality and can be stored until being used for manufacturing of components. In order to achieve the mechanical properties of the basic material test coupons were made via stacking of the prepregs and final sintering. With these input data a blade prototype including blade root was designed considering application relevant loads as rotation and aerodynamic resistance. The resulting design was optimized via finite element analysis including the anisotropic layered structure of the alloxide composites. As result a simple wedge design with a complex prepreg lay-up was chosen for a form fit of the blade root in a metallic collar. The fan blade was then produced from the prepregs and sintered in a one piece design. The mechanical proof test of the blade root fixation design was performed in a high velocity centrifuge up to failure at 4500 rpm. The results were evaluated and compared with the finite element prediction.

Topic: 4 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in composites : Oxides & Geopolymers (incl. short fibers)
Abstract no. 2061

# AN ORIGINAL CONCEPT FOR THE SYNTHESIS OF A COATING OR A COMPOSITE: THE FILM BOILING PROCESS


Presenting author: Dr MAILLé Laurence

Affiliation: University of Bordeaux - Laboratoire des Composites ThermoStructuraux
LCTS - 3 allée de la Boétie,33600 Pessac (France)

Co-authors: BESNARD Clémence (Laboratoire des Composites ThermoStructuraux), ALLEMAND Alexandre (CEA Le Ripault - LAboratoire des Composites ThermoStructuraux)



Nowadays, oxide/oxide composites are most of the time elaborated by sintering or CVI (Chemical Vapor Infiltration). This work focuses on an original and rapid process developed by the French Atomic Energy Commission (CEA): the film boiling chemical vapor infiltration. This technology works with a porous preform fixed to a carbon resistor or susceptor. This setup is immersed into a liquid precursor and heated above the precursor decomposition temperature. A film of vapor is created locally around the sample. The vapor decomposes inside the preform and leads to a densification. In this work, aluminium tri-sec butoxide, tetraethyle orthosilicate and barium isopropoxide were used to infiltrate alumina, silica and barium aluminosilicate in oxide preforms. Two experimental parameters have been studied: the heater intensity and time the processing.  A method to obtain the temperature by measuring the electrical resistance of the resistor has been developed. Microstructural analyses were carried out by environmental scanning electron microscopy (ESEM), X-ray diffraction (XRD) and high-resolution electron probe microanalysis (EPMA).

Topic: 4 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in composites : Oxides & Geopolymers (incl. short fibers)
Abstract no. 2758

# Innovative and differentiating technological platforms to bring CMCs at high TRL levels


Presenting author: Mme PAILLASSA Aude
Affiliation: IRT Saint Exupéry
Esplanade des Arts et Métiers,33405 TALENCE cedex (France)

Co-authors: PAILLASSA A. (IRT Saint-Exupéry Talence France), FERRES L. (IRT Saint-Exupéry Talence France)



Ceramic Matrix Composites are excellent candidates to replace metals and alloys in hot parts of aircraft engines. Their lower density and higher thermomechanical properties allow a better efficiency in order to respect increasingly stronger environmental and economic constraints. To reach these targets, different types of composites are developed from the SiC/SiC to the Oxide/Oxide. However, the cost of these materials might be prohibitive. Cost reduction can be achieved by using processes inspired from Organic Matrix Composites (OMC) manufacturing. Saint-Exupéry IRT is a joined development center enabling industrials and academics to work together to reach high Technological Readiness Levels. Since 2015, IRT has set up four differentiating platforms: three are dedicated to CMCs manufacturing and one to their characterization in engine environment.

RTM is one of the processes the most used with OMCs. A process derived from this one is used on our injection platform. Helped by companies specialized in tooling machining and manufacturing, we evaluate the scale up from single plane shape to multiple complex shape parts. This process can be used both for Oxide, Silicon Carbide or Carbon fiber reinforced Ceramics.

Another process inspired from OMCs is continuous tape impregnation line. We are working on both technologies: dry and wet impregnation for less expensive CMC manufacturing. Associated to this platform, we can develop and adapt slurries manufacturing up to 20l and control their physical characteristics (grain size, rheology, sedimentation rate).

The last production platform is dedicated to heat treatment and siliciuration. The heating device is multi-instrumented to enable accurate temperature control, to run under controlled atmosphere (1ry and 2ry vacuum or/with inert gas partial pressure) and to observe phenomenon occurring at elevated temperatures (videos and pictures).

Finally, to characterize under service these CMCs, we are setting up a fire test installation to evaluate thermal and mechanical behavior of demonstrators.

Topic: 4 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in composites : Oxides & Geopolymers (incl. short fibers)
Abstract no. 2073

# Formulation of oxide suspensions for liquid processing of ceramic matrix composites


Presenting author: M BOUTENEL Florian

Affiliation: Institut Clément Ader, Université de Toulouse, CNRS, IMT Mines Albi, INSA, UPS, ISAE-SUPAERO
Campus Jarlard,CT Cedex 09,81013 Albi (France)

Co-authors: AIMABLE Anne (Université de Limoges CNRS IRCER UMR 7315), DUSSERRE Gilles (Institut Clément Ader Université de Toulouse CNRS IMT Mines Albi INSA UPS ISAE-SUPAERO), CUTARD Thierry (Institut Clément Ader Université de Toulouse CNRS IMT Mines Albi INSA UPS ISAE-SUPAERO), CHARTIER Thierry (Université de Limoges CNRS IRCER UMR 7315)



This study deals with the formulation of oxide suspensions for liquid processing of ceramic matrix composites. Those processes are more advantageous than conventional ones regarding: cycle time, quality, flexibility and cost. To properly impregnate the fiber reinforcement, the suspension must be stable, well dispersed and have a low viscosity (< 1 Pa.s). Regarding those requirements, the dispersion of an alpha-alumina ultrafine powder (AKP50, d50 = 0.3 µm, Sumitomo Chemical) in aqueous suspension, using an ammonium polymethacrylate (Darvan® C-N, Vanderbilt Minerals) as dispersant, was investigated by zeta potential, sedimentation and rheological measurements. The dispersant concentration minimizing the viscosity was found to be 2.5·10-6 mol/m². Moreover, this concentration permits to shift the isoelectric point (IEP) from pH = 9.5 (without dispersant) to pH = 5.1. The influence of powder concentration on suspension viscosity is well described by a Krieger-Dougherty model. The maximum volume fraction was found to be equal to 47.6 vol.%, thanks to the viscosity. No significant sedimentation was observed, regarding the operating time of the process. This work comprises also the study of a commercial suspension of colloidal silica (Ludox AS-40, 40 wt.% suspension in water, W.R. Grace & Co.-Conn.). The zeta potential is negative from pH = 2 to pH = 11. The impact of dilution on viscosity was studied. For all powder loadings (from 1 to 40 wt.%), the viscosity is lower than 1 Pa.s (Newtonian) and the variation is also well described by a Krieger-Dougherty model. No sedimentation was observed. Furthermore, the mixture of both suspensions in a stoichiometric ratio of 3Al2O3-2SiO2, corresponding to mullite, was investigated. The natural pH and the IEP were measured at pH = 9.5 and pH = 3.0, respectively. The viscosity, as a function of solid concentration, is well described by a Krieger-Dougherty model. No significant sedimentation was observed.

Topic: 4 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in composites : Oxides & Geopolymers (incl. short fibers)
Abstract no. 2035

# Development of Ox/Ox composites for LPT components – Processing and Properties


Presenting author: M NISHIKAWA Kosuke

Affiliation: Mitsubishi Heavy Industries, Ltd.
,1200, higashi tanaka,485-8561 komaki (Aichi, Japan)

Co-authors: KURIMURA Takayuki (Mitsubishi Heavy Industries Ltd.), MATSUMOTO Mineaki (Mitsubishi Heavy Industries Ltd.), SEKIGAWA Takahiro (Mitsubishi Heavy Industries Aero Engine Ltd.), FUKUSHIMA Akira (Mitsubishi Heavy Industries Aero Engine Ltd.), NOGAMI Ryoma (Mitsubishi Heavy Industries Aero Engine Ltd.), TAMUGI Azusa (Mitsubishi Heavy Industries Aero Engine Ltd.), USHIDA Masanori (Mitsubishi Heavy Industries Aero Engine Ltd.), KAKISAWA Hideki (National Institute for Materials Science (NIMS), HASEGAWA Yoshio (ART KAGAKU Co. Ltd), KUMETA Kazuhiro (NITIVY CO. LTD.)



Future gas turbine engines are aimed for higher efficiency with lower exhaust emissions. Efficiency of the gas turbine engine increases with an increase in turbine inlet temperature (TIT). Current Nickel based super-alloys are operating at their maximum temperatures and cannot withstand an increase in temperature. Ceramic matrix composites (CMCs) are one of the best candidate materials because of their low weight and high temperature capability. In order to successfully implement CMCs in gas turbine engines, the cost affordable and environmental superior Ox/Ox composites by Polymer Infiltration and Pyrolysis (PIP) using ceramic precursor have been developing. PIP has advantage as lower temperature than conventional sintering process such as the hot press process and the degradation of the fiber can be suppressed. On the other hand, if the fiber makes contact with PIP ceramic precursor during pyrolysis, the degradation of the fiber would be occurred. In order to solve this issue, we focus on the interface coating such as fugitive coating. Ox/Ox composites which changed several parameters such as fiber coating and matrix slurry etc. were manufactured and evaluated. As a result, Ox/Ox composites with stable material properties could be developed by optimizing the order of the process atmosphere to suppress the disappearance of the carbon interface coating during the manufacturing process. We will discuss the relationship between process parameters and material properties of these composites.


This research was supported by the Structural Materials for Innovation of the Cross ministerial Strategic Innovation Promotion Program (SIP) of Japan Science and Technology (JST).

Topic: 4 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in composites : Oxides & Geopolymers (incl. short fibers)
Abstract no. 1864

# Development of Ox/Ox composites for LPT components – mechanical properties


Presenting author: M FUKUSHIMA Akira

Affiliation: Mitsubishi Heavy Industries Aero Engine, Ltd.
1200, Higashi-Tanaka,485-0826 Komaki (Aichi, Japan)

Co-authors: SEKIGAWA Takahiro (Mitsubishi Heavy Industries Aero Engine Ltd), NOGAMI Ryoma (Mitsubishi Heavy Industries Aero Engine Ltd.), TAMUGI Azusa (Mitsubishi Heavy Industries Aero Engine Ltd.), USHIDA Masanori (Mitsubishi Heavy Industries Aero Engine Ltd.), KOSUKE Nishikawa (Mitsubishi Heavy Industries Ltd.), KURIMURA Takayuki (Mitsubishi Heavy Industries Ltd.), MATSUMOTO Mineaki (Mitsubishi Heavy Industries Ltd.), KAKISAWA Hideki (NIMS), HASEGAWA Yoshio (Art Kagaku Co. Ltd.), KUMETA Kazuhiro (Nitivy Co. Ltd.)



For the higher efficiency of the aircraft engines, light-weight and high heat resistance materials are required. One of the answers is to use the ceramic matrix composites, CMCs, instead of the superalloys, conventional heat resistance metals. Today GE has already applied the SiC/SiC CMCs for the high pressure turbine components in the commercial engines. On the other hand, the cost affordable and environmental superior Ox/Ox composites are developing for the lower temperature and the lower stress components. In this article, the mechanical properties of Ox/Ox composites, which are developed in Japan, for the low pressure turbine of the aircraft engines are evaluated. The materials and processing details are showed in other article. For high temperature evaluation, the digital image correlation system using ultra violet light is applied. The mechanical properties, such as tensile, fatigue and notch intensity, are investigated. Tensile tests are evaluated at RT and 1100C in air. The stress controlled low cycle fatigue tests are evaluated at RT and 1000C with R=0.1 in air. The notch intensities are evaluated using the double edge notch test specimen at RT. Composite microstructure, as well as damage and failure mechanisms were investigated.


This research was supported by the Structural Materials for Innovation of the Cross ministerial Strategic Innovation Promotion Program (SIP) of Japan Science and Technology (JST).

Topic: 4 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in composites : Oxides & Geopolymers (incl. short fibers)
Abstract no. 1989

# Fibre-reinforced oxide ceramics with integrated CNT network produced by ceramic injection moulding

Presenting author: Mme BöTTCHER Maike
Affiliation: Department of Lightweight Structures / Polymer Technology, Chemnitz University of Technology
,09107 Chemnitz (Germany)

Co-authors: NESTLER Daisy (Department of Lightweight Structures / Polymer Technology Chemnitz University of Technology), RAMALINGAME Rajarajan (Professorship of Electrical Measurements and Sensor Technology Chemnitz University of Technology), RAJENDRAN Dhivakar (Professorship of Electrical Measurements and Sensor Technology Chemnitz University of Technology), WENDLER Frank (Professorship of Electrical Measurements and Sensor Technology Chemnitz University of Technology), KANOUN Olfa (Professorship of Electrical Measurements and Sensor Technology Chemnitz University of Technology), KROLL Lothar (Department of Lightweight Structures / Polymer Technology Chemnitz University of Technology)

Ceramic materials are suitable for use in the high temperature range. Oxide ceramics, in particular, have a high potential for long-term applications under thermal cycling and oxidising atmosphere. However, monolithic oxide ceramics are unsuitable for use in high-temperature technical applications because of their brittleness. Thin-walled, oxidation, and high-temperature resistant materials can be developed by reinforcing oxide ceramics with ceramic fibres such as alumina fibres. Of outstanding importance is the increase in the mechanical stability of the composites in comparison to the non-fibre reinforced material. Possible stresses or cracks can be derived along the fibre under mechanical stress or deformation.
Components made of fibre-reinforced ceramic composites with oxide ceramic matrix (OCMC) are currently produced in manual and thus price-intensive processes for small series. In order to be able to realise mass production, the injection moulding process (CIM - Ceramic Injection Moulding) established in the manufacture of monolithic oxide ceramics was used for the production of fibre-reinforced oxide ceramics. In addition, the OCMCs were functionalised. Carbon nanotubes (CNTs) were integrated into the material as electrically conductive components. The formation of a CNT network leads to an electrical conductivity. This provides the possibility to achieve process monitoring through the functionalised high-temperature material.
The production of a fibre-reinforced oxide ceramic with integrated CNT network was realised over a number of process steps. These include dispersing the CNTs, introducing the CNTs into the oxide ceramic matrix material and adding the fibre component in the compounding process.

This is followed by the injection moulding process and the debinding and sintering of the specimens. The influence of the CNT content has been investigated by the production and characterisation of test specimens with variation of the CNT content. For this purpose, microstructure investigations were carried out, mechanical-static tests were performed and the electrical conductivity was examined by impedance spectroscopy.

Topic: 4 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in composites : Oxides & Geopolymers (incl. short fibers)
Abstract no. 2190

# Evolution of the rheological behaviour of an aqueous suspension of geopolymeric precursors during "maturation"


Presenting author: Dr DUSSERRE Gilles

Affiliation: Institut Clément Ader (ICA), Université de Toulouse, CNRS, Mines Albi, UPS, INSA, ISAE-SUPAERO
Campus Jarlard,81013 Albi CT Cedex 09 (France)

Co-authors: FARRUGIA Anaïs CUTARD Thierry (Université de Toulouse CNRS Mines Albi UPS INSA ISAE-SUPAERO)



Aqueous suspensions of geopolymeric precursors are promising ways to produce glass-ceramic matrix composites by conventional liquid composite moulding processes (slurry infiltration). However, the rheological behaviour is complex and not necessarily suitable for such processes because of high viscosity values. A long "maturation" step of the suspension is therefore necessary to obtain a Newtonian behaviour, and the amount of water must be optimized to reach the desired viscosity without reducing drastically the solid fraction. This work investigates the changes in rheological behaviour of an aqueous suspension of geopolymeric precursors during maturation at -18°C. A phenomenological model is proposed to describe the effect of the "maturation" step on the rheological behaviour in the processing temperature range, before the onset of polymerization, and accounts for dilution with water. The model assumes a population of large particles (up to 1 ?m) suspended in an aqueous suspension of fine particles. A full-scale separation between both particle populations allows expressing the viscosity of the suspension as the product of two Krieger-Dougherty models. "Maturation" is schematized as the dissolution of large particles into the suspension of fine particles, which is assumed to be Newtonian. A thixotropic model, acting on the effective solid fraction of large particles, is identified and results in a proper description of the viscosity of the suspension at equilibrium between 10-2 s-1 and 200 s-1, as well as during destructuration experiments at 70 s-1.

Topic: 4 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in composites : Oxides & Geopolymers (incl. short fibers)
Abstract no. 2374

# Reaction assisted flash sintering of Al2O3-YAG ceramic composites with eutectic composition


Presenting author: Prof LIU Jinling
Affiliation: Southwest Jiaotong University
111 North 1st Section,2nd Ring Road,610031 Chengdu (Sichuan, China)

Co-authors: LIU Dianguang (Southwest Jiaotong University), XU Xiang (Southwest Jiaotong University)



We report that the Al2O3-Y2O3 ceramic composites with eutectic composition can be flash sintered at 1250 oC under the applied electrical field of 550 V/cm in an extremely short time (30 sec). The solid-state reactions between Al2O3 and Y2O3 facilitated the flash sintering process. The sintered Al2O3-YAG ceramic composites exhibit relatively high hardness and high fracture toughness. These results demonstrate the feasibility of employing the flash sintering technique to fabricate oxide ceramic composites.

Topic: 4 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in composites : Oxides & Geopolymers (incl. short fibers)
Abstract no. 2448

# Valorization of ceramics industry waste: elaboration and characterization of a new refractory composit acid based geopolymer/ vitreous ceramic breakages


Presenting author: Dr MAJDOUBI HICHAM
Affiliation: faculté des sciences ben m'sik univérssité hassan II casablanca
Faculté des Sciences Ben M'Sik,Av Driss El Harti Othmane,20670 casablanca (faculté, Morocco)
Co-authors:



This study consists in the elaboration and characterization of a refractory composite of acid-based geopolymers / vitreous ceramic breakages.
The experimental process consists in the addition of vitreous ceramic breakages to the acidic geopolymer leg as reinforcement with different percentages and granulometries in order to increase the thermal and chemical mechanical performance of the geopolymer. Compressive strength, high temperature resistance and resistance to aggressive environments are studied
Preliminary results have shown that the addition of a percentage of vitreous ceramic breakages between 5% and 50% can increase compressive strength up to 130 MPa, as well as the elaborated composites can reach 1700°C without any sign of fusion, which indicates the possibility of using them as refractories, The next step in this process is to study the resistance to aggressive environment of the elaborated composites such as basic and marine acid environments.
In the light of these results, it can be deduced that geopolymer/ vitreous ceramic breakages refractory composites can recover waste from the ceramic industry and reduce the impact on the environment.

Topic: 4 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in composites : Oxides & Geopolymers (incl. short fibers)
Abstract no. 2102

# Evaluation of mechanical properties of Al2O3/Al2O3 using fiber bundle composites


Presenting author: M YAMAMOTO Shohei

Affiliation: Tokyo University of Science
3-1-1-1859,Yoshinodai, Chuo-ku,252-5210 Sagamihara city (Kanagawa Prefecture, Japan)

Co-authors: GOTO Ken (JAXA), KOGO Yasuo (Tokyo University of Science), INOUE Ryo (Tokyo University of Science)



In this study, the mechanical properties of Al2O3 fiber reinforced Al2O3 ceramics (Al2O3/Al2O3) were evaluated by using fiber bundle composites. The Al2O3 fiber bundles used were Al2O3–SiO2 polycrystalline fiber (Nichibi ALF, R-350FK-D510). The number of fibers in bundle is 320 and the nominal diameter of fiber is 20?m. Carbon coating on Al2O3 fiber surface as weak interface was prepared using phenol resin diluted with ethanol and 1wt.% carbon black. Bundle composites were fabricated by infiltration by ?-Al2O3 slurry into Al2O3 fiber bundles. After drying at 90? for 6 hours, bundle composites were sintered at 1150? in Ar atmosphere for 2 hours. As the evaluation method, the bundle composite tensile strength, dry bundle strength by single fiber tensile test and mechanical properties of fiber matrix interface were obtained.?The tensile strength (?) was calculated by dividing the fracture load by the cross sectional area of bundle composites, and the fiber efficiency (Y) was calculated based on maximum strain of Al2O3 fiber bundle FRP. Dry bundle strength was estimated from Colman's equation and compared with bundle composites. The fiber volume fraction of bundle composites was approximately Vf=55% and the composite porosity was approximately 26%. The tensile strength and fiber efficiency of Al2O3/Al2O3 were approximately ?=309MPa and Y=45%, and these of Al2O3/C/Al2O3 were ?=410MPa and Y=65%. The value obtained by converting the dry bundle strength to the composites strength was 380MPa, and only the Al2O3/C/Al2O3 were showed higher strength than dry bundle strength.

Topic: 4 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in composites : Oxides & Geopolymers (incl. short fibers)
Abstract no. 2807

# Solutions for CMC

Presenting author: Mme MARRA Livia
Affiliation: BAIKOWSKI
1046 route de Chaumontet,74330 POISY (France)

Co-authors:

The ceramic fibers used in CMCs have a typical diameter of around 10 µm. The matrix precursors must be much smaller than this length scale (<<10 µm) for three reasons : easy processability (intimate mixing during CMC fabrication) ; low sintering temperature and good mechanical properties after sintering.
With its broad range of nanoparticles, Baikowski is a leading supplier of matrix precursors for Oxide/Oxide CMCs.
The poster will review the properties of the oxide powders suitable for the matrix in Oxide/Oxide CMCs : tight and monomodal particle size distribution, high sintering reactivity, high chemical purity…
Baikowski is a partner of choice for customized solution: Powder suspension; Specific surface; Repairable CMC.

# Topic: 5

# Innovative Design, Advanced Processing, and Manufacturing Technologies processing in non-oxide composites: SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 1854

# SiC ceramic matrix composites with high load-carrying ability under complex stress conditions


Presenting author: Prof CHENG Laifei

Affiliation: Northwestern Polytechnical University
127 West Youyi Road,Beilin District,710072 Xi'an (Shaanxi, China)

Co-authors: YE Fang (Northwestern Polytechnical University), GAO ZhiWei (Northwestern Polytechnical University), CHEN NaiQi (Northwestern Polytechnical University)



Silicon carbide (SiC) ceramic matrix composites have the advantages of light weight, high strength, high temperature resistance and oxidation resistance, and have broad application prospects in the aerospace field. Continuous fiber toughened ceramic matrix composites (CFCCs) have metal-like fracture behavior, which are not sensitive to cracks and do not cause catastrophic damage. CFCCs can achieve the best effect of strengthening and toughening, and the fracture toughness can be as high as 20MPa?m1/2. However, the fiber preforms of CFCCs are usually two-dimensional (2D), two and a half-dimensional (2.5D) or three-dimensional (3D) woven structures, leading to their anisotropic characteristics. Besides, CFCCs with the weak fiber/matrix interface also have the low proportional ultimate stress due to the non-linear mechanical behavior. When CFCCs are used to prepare complex structural parts in complex stress environments, these shortcomings may reduce the reliability of the components. In order to solve this problem, SiC whiskers toughened SiC ceramic matrix composites (SiCw/SiC composites) with isotropic characteristics, high proportional ultimate stress, strength and toughness were prepared from the point of composite structure design by means of reinforcement (whisker and nanowire) structure design, interface bonding strength control and matrix modification. The SiCw/SiC composites prepared in this paper have a bulk density of 3.0g?cm-3, porosity of less than 5%, excellent mechanical properties such as fracture toughness up to 8.0MPa?m1/2 and bending strength up to 425MPa. The developed preparation processing of SiCw/SiC composites can realize near-net shape molding. It is expected to have a service temperature range of 1300~1500°C. SiCw/SiC composites can be considered as an effective supplement for CFCCs, and expand the application as high temperature structural materials.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 2270

# Physically Augmented Hybrid Virtual Test: an NDE Methodology for Accelerating CMC Development.

Presenting author: Prof SHICHIJO Naohiro
Affiliation: Tokyo University of Technology
1404-1 Katakura,192-0982 Hachioji (Tokyo, Japan)

Co-authors: SATO Mitsuhiko (Tokyo University of Technology), TANAKA Yoshihisa (Tokyo University of Technology), FUJIWARA Chikara (Tokyo University of Technology), HATTA Hiroshi (Tokyo University of Technology), OOKUBO Tomomasa (Tokyo University of Technology), FURUI Mitsuaki (Tokyo University of Technology), GOTO Ken (Institute for Space and Astronautical Science), SATO Eiichi (Institute for Space and Astronautical Science)

Simulating degradation of Ceramic Matrix Composite's (CMCs) is one of the biggest challenges in computational material science due to its complicated microstructure and constitutive phenomena, especially in their working conditions. Nevertheless, the need for simulating CMCs is ever increasing due to increased usage as airplane turbine engines parts and remaining high cost for fabrication and testing. To cope with this, we introduce a new concept “Augmented virtual testing”, a hybrid assessment approach of CMC degradation using both virtual testing and physical testing. By controlling conditions of physical testing dynamically, parameters in degradation model of each constitutive elements are precisely obtained. By using those parameters simulation would be accelerated compared to pure virtual testing. As our first report in augmented virtual testing, basic concept and theoretical backgrounds are presented.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 1907

# Novel Non-oxide CMCs: MAX phase/SiC fiber Composites

Presenting author: Prof GONZALEZ-JULIAN Jesus
Affiliation: Forschungszentrum Jülich
Wilhelm-Johnen-Straße,52425 Jülich (Germany)

Co-authors: DASH Apurv (Forschungszentrum Jülich), MALZBENDER Jürgen (Forschungszentrum Jülich), GUILLON Olivier (Forschungszentrum Jülich), VASSEN Robert (Forschungszentrum Jülich)

MAX phases are a new family of high temperature materials with general formula of $M_{n+1}AX_n$, where M corresponds with an early transition metal, A is a A-group element, X is C or N, and n is typically 1, 2 or 3. They are excellent candidates for high temperature application (> 1000 °C) due to their unique combination of properties, bridging the gap between ceramics and metals. As ceramics, they are lightweight, and show excellent oxidation and corrosion resistances, meanwhile as metals, they present good damage tolerance, and high thermal and electrical conductivities. Nevertheless, better mechanical properties are required to transfer MAX phases into the market, but development of MAX phase composites has been slightly investigated.
In this work, $Cr_2AlC$ and $Ti_3SiC_2$ were synthesized, mixed with 5 and 10 wt.% of SiC fibers, and sintered by Spark Plasma Sintering. Creep resistance was characterized as function of fiber content, temperature, and stress, meanwhile the tribological response under different loads was measured. Incorporation of fibers increases more than one order of magnitude the creep resistance, as well as reduces the wear rate. Furthermore, injection molding of MAX phase composites will be presented, showing the possibility to transfer these materials to industry.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 2160

# INNOVATIVE LOM MANUFACTURING ROUTE FOR HIGH-PERFORMANCE CYLINDRICAL-SHAPED OBJECTS


Presenting author: Dr CHIODI Mirco
Affiliation: Petroceramics spa
viale Eurolpa 2,24040 Stezzano (Italy)
Co-authors: VALLE Massimiliano (Petroceramics spa)



Laminated composites made of Carbon Fibers (CFs) dispersed in ceramic matrices have been used for high-demanding applications (aerospace, wind power energy or braking systems) for decades. Among all the su-perior characteristics of composites compared to traditional, isotropic materials, three are the most well-known. They possess high-specific strength (strength-to-mass ratio), high thermal conductivity (especially at high temperatures) and extreme overall resistance in harsh environments. Such attracting properties largely depend on the possibility to precisely control the CFs architecture geometry. Current standard Laminated Object Manufacturing (LOM) routes only allow a limited control over the fibers internal positioning, especially for short fibers which need long, expensive and even manual-positioning steps.

Recently, we introduced a simple and fast approach to manufacture laminated objects having a fine control over short fibers positioning inside a composite. Instead of super-imposing a series of sheets to be moulded, our novel technique (ROLL-IT) relies on the controlled winding of a carrier sheet on which short CFs are dispersed. This approach naturally imposes a tangential directionality to virtually all the short fibers in the final product. Thus, ROLL-IT is straightforwardly applied to any object having a cylindrical shape or a central symmetry axis. Winding on a mandrel, we can also control the degree of tension imposed to the ensamble, tailoring the final structure of the green body. The sheet can be feeded with additives or reinforcing elements before the final winding. In case of a pre-preg, the green body can also be heated to cure/polymerize during the winding process without the need of the subsequent impregnation steps typical of standard processes like filament winding.

Here we will introduce ROLL-IT discussing a case of study in which we exploited its peculiarities to produce high-performance, carbon-based composite brake discs in a faster and cheaper way compared to other standard manufacturing processes.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 2000

# Thermodynamic and kinetic of liquid metal infiltration in TiC-SiC or SiC porous compacts

Presenting author: Dr ROGER Jérôme
Affiliation: LCTS - Université de Bordeaux - CNRS
3 allée de La Boétie,33600 PESSAC (France)
Co-authors: SALLES Marie (Laboratoire des Composites ThermoStructuraux (LCTS)

The elaboration of TiSi2/SiC composites was considered by capillary infiltration of liquid silicon or Si-Ti molten alloys in SiC of SiC+TiC compacts in order to identify the thermodynamic and kinetic limitations. Preliminary thermodynamic calculations were performed in the Ti-Si-C system with CALPHAD methodology to select the compositions of the liquids and the operating temperatures. Three cases were chosen: 1) infiltration of molten TiSi2 in pure SiC compacts at 1550°C, 2) reactive infiltration of pure molten silicon in SiC+TiC compacts at 1450°C, 3) reactive infiltration of the eutectic Ti0.16Si0.84 alloy in SiC+TiC compacts at 1380°C. The compacts were prepared from mixtures of micronic SiC (? or ? polytypes) and TiC powders, then strengthened by heating at 1500°C for 1 h under high vacuum. The mixtures compositions were chosen to fill totally the porosity of the compacts of about 50% or with an excess of TiC. The infiltration of the compacts was performed without and with controlling the contact between the liquid and the compact. The weight gain during the infiltration was measured in the former case. The depth of the infiltration fronts, the phases present and their proportions in the samples were estimated from backscattered-electron (BSE) micrographs and image analyses. Experimental results evidenced that the interactions between the liquid and the powders composing the compacts, SiC with and without TiC, are complex. The obtained materials differ from the expected composites that are generally not dense and contain variable quantities of free silicon. These experimental results are explained by thermodynamic calculations. This work proves that activity gradients play a determining role during the infiltration process by initiating the dissolution and the diffusion of atoms in the liquids. The elaboration of SiC-TiSi2 composites is complex but could be possible in limiting or in prohibiting the activity gradients. Finally, some improvements are proposed.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 2207

# Study of the densification of SiC matrix by film boiling process

Presenting author: Dr BONNAMY Sylvie
Affiliation: CNRS/Université d'Orléans
ICMN,1B, rue de la Férollerie,45071 ORLEANS cedex 2 (France)

Co-authors: SERRE Aurélie (CEA DAM Le Ripault 37260 Monts France), AUDUBERT Fabienne (CEA Cadarache 13108 Saint-Paul-Lez-Durance France), BLEIN Joëlle (CEA DAM Le Ripault 37260 Monts France), BRUNETON Eric (CEA DAM Le Ripault 37260 Monts France), DAVID Patrick (CEA DAM Le Ripault 37260 Monts France)

Ceramic Matrix Composites can be manufactured by different ways and the one studied in this work is the film-boiling process. The film-boiling process is a particular chemical vapour infiltration process for the densification of carbon and other ceramic matrices. The densification starts from a porous preform, heated at high temperature in a bath of liquid precursor which becomes gaseous in contact with hot surfaces. The cracking of the produced vapours results in a solid deposit constituting the matrix of the final composite. The main advantage of the film-boiling technique is its high densification rate: up to several ?m/h depending on the experimental conditions (versus ~ 2?m/h in CVI). To get a better insight on this process, a laboratory-made equipment, a mini-kalamazoo, was developed, set-up and adapted to the needs of our study.
The first tests were done with the methyltrichlorosilane (MTS) precursor widely used for SiC CVD/CVI. Characterizations of the deposits (density, homogeneity, crystalline phase…) in using analytical techniques, such as XRD, EDS/SEM, EPMA, HR-TEM, showed the formation of SiC but also the occurrence of carbon. Pure SiC can be locally obtained at the beginning of the film-boiling process in some specific experimental conditions. For most of the experiments, the use of pure MTS as precursor leads inevitably to the formation of free carbon in the SiC deposit. Several improvement routes were proposed and tested to remove this carbon excess. Some of the efficient and promising routes consisted in the use of MTS mixed with a silicon precursor free of carbon and the use of two non-chlorinated SiC precursors: CVD 4000 liquid precursor and hexamethyldisilane. The deposit growth rates are significantly higher with the film-boiling process compared to the classical processes. All the data show that the film-boiling process is promising for the manufacturing of SiC and new carbide materials.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 1848

# Properties of SiC-SiBCN matrix composite prepared by CVI plus PDC method


Presenting author: Prof LUAN Xingang

Affiliation: Northwestern Polytechnical University
Youyixi Road 127,710072 Xi'an (Shaanxi, China)

Co-authors: ZOU Yun (Northwestern Polytechnical University), WANG Lei (Northwestern Polytechnical University), XU Xinming (Northwestern Polytechnical University), RIEDEL Ralf (TU Darmstadt), CHENG Laifei (Northwestern Polytechnical University)



A new method of Chemical Vapor Infiltration (CVI) combined with Polymer Infiltration and CVI Online Pyrolysis (PIOP) is proposed to fabricate C/SiC and SiC/SiC with SiBCN self-healing matrix which is polymer derived ceramics (PDC) of PSNB precusor. The microstructural evolution during the densification of porous C/SiC preform through CVI+PIOP method, traditional CVI+PIP method and single CVI method was studied. The oxidation behaviors of three kinds of composites were studied in air at different temperatures. Oxidation behaviors of CVI+PIOP C/ (SiC-SiBCN)x and SiC/(SiC-SiBCN)x were also studied in wet-oxygen atmospheres at different temperatures. Oxidation resistant mechanisms of CVI+PIOP sample were discussed.High temperature creep behavior and mechanisms of SiC/(SiC-SiBCN)x was investigated in a wet-oxygen atmosphere further.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 1936

# FABRICATION AND PROPERTY EVALUATION OF ACTIVE FILLER INCORPORATED CERAMIC MATRIX COMPOSITE


Presenting author: M VIJAY Vipin
Affiliation: VIKRAM SARABHAI SPACE CENTRE
CERAMIC MATRIX PRODUCTS DIVISION,ASCG/PCM/VSSC/ISRO,695022
THIRUVANANTHAPURAM (KERALA, India)
Co-authors:



2D Cf/SiC CMC, with & without filler and interphase materials were fabricated via TiSi2 controlled polymer pyrolysis. Flexural strength and oxidation resistance property of all the CMCs after a single PIP cycle were investigated and correlated. Presence of filler and interphase material exhibited a vital role in the properties of a CMC. Presence of filler in an interphase free CMC exhibited deteriorates its flexural strength due to the detrimental reaction with carbon fibre reinforcement. The sacrificial interphase (PyC) and inert interphase (BN) improved the mechanical property of the CMC by protecting the fibres from the attack of the TiSi2 active filler. BN interphase incorporated CMC exhibited the highest mechanical property (45.8±9.52 MPa) after single PIP cycle, owing to the inert nature of the interphase material. Cf/SiC and Cf/SiC-TiSi2 CMCs exhibited better oxidation resistance properties among filler free and filler incorporated CMCs respectively.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 1904

# Novel High-Performance C/C-SiC Composites Based on Cellulose Precursors


Presenting author: Mme SCHNECK Tanja

Affiliation: Deutsche Institute für Textil- und Faserforschung (DITF)
Körschtalstraße 26,73770 Denkendorf (Germany)

Co-authors: "DR. HERMANUTZ Frank ( Deutsche Institute für Textil- und Faserforschung (DITF)), PROF. BUCHMEISER Michael R. ( Deutsche Institute für Textil- und Faserforschung (DITF); Institut für Polymerchemie, Universität Stuttgart)



Carbon fiber- (CF) reinforced silicon carbide (C/C-SiC) composites are ceramic materials with high performance properties. During the manufacturing process of C/C-SiC composites, CFs are first embedded in a polymer matrix resulting in CF-reinforced polymers (CFRP), then carbonized and finally infiltrated with liquid silicon. Currently, the polymer matrices are crude oil based. For sustainability, wood-based precursors are used but so far not competitive with state of the art C/C-SiC composites.

New C/C-SiC composites based on cellulose reinforcement fibers embedded in a pure cellulose matrix (ACCs) were manufactured.[1] For the formation of the cellulose matrix, pulp was dissolved in different concentrations in an ionic liquid (IL). Technical cellulosic fibers were embedded therein using standard composite technologies. For C/C-SiC production, the all-cellulose composites (ACCs) were carbonized and infiltrated with liquid silicon.[2] The carbon yield could be significantly increased with a carbonization aid. The microstructure and mechanical properties of ACCs and C/C-SiC composites were influenced by the carbonization aid and by the cellulose concentration of the cellulose-matrix forming dope. Properties of the semi-finished goods and end products were characterized by scanning electron microscopy (SEM) and three-point bending tests, respectively. Further, different shapes of ACCs (tubes, Z-profiles) were manufactured to obtain shaped C/C-SiC composites directly by an easy and uncomplicated manufacturing process.



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Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 1625

# Molds local multi-instrumentation to describe the growth of ceramic green bodies during a slurry cast process : correlation with the process parameters and comparison with 1D model


Presenting author: Dr EBERLING-FUX Nicolas
Affiliation: Safran Ceramics
105 avenue Marcel Dassault,33700 Mérignac (France, France)
Co-authors: ROS William (Safran Ceramics)



Injection molds have been instrumented using local pression and resistive sensors in order to locally describe the growth of ceramic green bodies during an injection / filtration process of ceramic suspensions. The signal analyses can be successfully correlated to the injection parameters (pression, injected volume, flow) and leads to the understanding of the green matrix formation mechanisms for monolitic ceramics and ceramic matrix composites. The different results are then compared with the predictions of the classical Ruth filtration model.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 1867

# Preparation Evaluation and Combustion Rig Tests of an Effusive Cooled SiC/SiCN Panel

Presenting author: Mme HöNIG Sandrine
Affiliation: (1) German Aerospace Center (DLR), Institute of Structures and Design (BT)
Pfaffenwaldring 38-40,70569 Stuttgart (Germany)

Co-authors:

SiC/SiCN ceramic matrix composites (CMCs) are promising candidates for components of aero-engines. To evaluate the properties of these CMCs under realistic conditions, a quasi-flat panel with effusion cooling holes was investigated in a high pressure combustor rig. A Tyranno SA3 fabric based SiC/SiCN composite with high strength and fracture strain was manufactured via polymer infiltration and pyrolysis (PIP) process. Due to its weak matrix no fiber coating was necessary for damage tolerant behaviour. The cooling holes in the panel were introduced via laser drilling. An outer coating of CVD-based SiC was finally applied for enhanced oxidation resistance.
The specimen was tested in the combustor rig. Temperature distributions and cooling effectiveness were evaluated. The macrostructure and coating quality were evaluated via CT scans before and after the combustor test. Local microstructure modifications were observed after laser drilling and coating. No crack formation was observed in the CMC panels.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 2230

# Crack Trapping Mechanisms in Discontinuous Carbon Fiber Carbon Matrix Composites


Presenting author: Prof KAGAWA Yutaka
Affiliation: Tokyo University of Technology
1404-1,Katakura,192-0982 Hachioji (Tokyo, Japan)

Co-authors: HATTA Hiroshi (Tokyo University of Technology)



Microfracture behavior of discontinuous carbon fiber-SiC matrix composites play an important role on entire damage progress because the composite allows accumulation of microcracks. Heterogeneous microstructure of the composite allows various kinds of crack microstructure interactions. In the present study, major attention has been focused on the interaction process of cracks in Si or SiC phase and discontinuous carbon fiber-SiC/C minicomposites in the composite. Experiential observation of the interaction process and numerical analysis of the interaction process are carried out including anisotropic behavior of used carbon fiber. These results reveal that microfracture event is mostly controlled by thermal stress and microcrack stability is controlled by interface debonding process, which usually takes place before crack arrives at interface between Si/SiC phase and the minicomposite phase. Discussions are made on stress/strain fields and crack trapping process at the interface.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 2247

# Effect of the spacing of diffusion-assisting holes on the mechanical properties of a thick-section 3D needled C/SiC composite fabricated by machining-aided chemical vapor infiltration


Presenting author: Dr ZHANG Jiaxin

Affiliation: Science and Technology on Thermostructural Composite Materials Laboratory, Northwestern Polytechnical University
127 West Youyi Road,Beilin District,710072 Xi'an (Shaanxi, China)

Co-authors: CHENG Laifei (Science and Technology on Thermostructural Composite Materials Laboratory Northwestern Polytechnical University), LIU Yongsheng (Science and Technology on Thermostructural Composite Materials Laboratory Northwestern Polytechnical University), ZHAO Hui (b Science and Technology on Thermostructural Composite Materials Laboratory Northwestern Polytechnical University), ZHANG Yi (Science and Technology on Thermostructural Composite Materials Laboratory Northwestern Polytechnical University), WANG Jing (Science and Technology on Thermostructural Composite Materials Laboratory Northwestern Polytechnical University), HE Zongbei (State Key Laboratory for Nuclear Fuel and Materials Nuclear Power Institute of China)



Machining-aid chemical vapor infiltration (MACVI), a modified chemical vapor infiltration (CVI), is the process of using diffusion-assisting holes to increase gas transport and thus matrix deposition in the middle layers of thick-section ceramic matrix composites (CMCs). In MACVI, the spacing of the diffusion-assisting holes (Sh) is an important parameter, which affects the final mechanical properties of the fabricated composites. Here, the effect of Sh on the mechanical properties of a 10-mm-thick 3D needled C/SiC (3DN C/SiC) is investigated. Results showed the decrease of Sh led to an increased strength of the composite. By decreasing the Sh from 8 mm to 5 mm, the corresponding flexural strength increased from 274±49 MPa to 325 ± 33 MPa.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 1932

# Material removal mechanisms in machining of Ceramic Matrix Composites

Presenting author: M GARCIA LUNA Gonzalo
Affiliation: Rolls-Royce UTC at University of Nottingham
Advanced Manufacturing Building C47,522 Derby Road,NG7 2RD Nottingham (Nottinghamshire, United Kingdom)
Co-authors:

Ceramic Matrix Composites (CMCs) have been identified as a key material system for improving aero engine performance as they offer low density, high strength and stiffness, and superior environmental resistance at high temperatures. CMC parts are usually manufactured near-to-net-shapes, so only surface finishing operations and machining of some detailed features are required, but due to its heterogeneous, hard and brittle nature, these materials are considered within the most difficult-to-machine. Understanding the material removal mechanism and optimum cutting tool geometry when machining CMCs, is a critical enabler for achieving high component quality at highest efficiency and minimum cost.

The paper reports on the research focused on the material fracture mechanisms under the machining conditions of CMCs with single cutting edges of different characteristics, such as shape and edge radii. In order to reproduce the machining processes kinematics, high-speed scratch tests have been carried out with different cutting edge geometries on an adapted Charpy pendulum to determine the influence on the surface integrity. The fracture characteristics of the different CMC material constituents have been assessed regarding the direction of the scratch with respect to the orientation of fibres. Moreover, inspection of the ‘scratched’ surface for the different cutting edge feature geometries implies a change in the mechanisms governing the material removal process.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 1865

# 1200-1800°C High Temperature Bending Behavior of C/C-SiC Composites Fabricated Via Reactive Melt Infiltration


Presenting author: M WU Xiaojun

Affiliation: Xi'an Aerospace Composite Materials Research Institute
shanxi,xian,710025 xian (China)

Co-authors: WANG KUNJI



A low cost method for the preparation of thermal structure C/C-SiC composites was explored by means of reactive melt infiltration (RMI), and the silicon infiltration behavior, and bending behavior at 1200-1800? high temperature of the composites were studied in this research. Firstly, the C/C porous preforms were prepared by CVI+PIC methods. The capillary infiltration test of C/C porous preforms was carried out using water instead of liquid silicon. The results show that the permeability law of water to C/C porous preforms is basically similar to that of liquid silicon, and the capillary permeability rate tends to decrease with the increase of the permeability height. The sample shows the morphology of high ends and low middle before infiltration, indicating that the permeability rate in the edge region is faster than that in the middle region. The SiC matrix formed by RMI has a network distribution in the composite, and the pore distribution is uniform. The pore size range estimated by ?-CT is 0~0.13mm, in which the pore size of the small pore(less than 30?m) is about 84%. The micro pores are mainly located in the fiber bundle. With the increase of temperature(1200?~1800?), the bending fracture strain of C/C-SiC composites decreased first and then increased. The fracture strain decreased from 0.85 at 1200? to 0.77 at 1400?, and then slowly increased to 0.97 at 1800?. However, the bending strength is opposite to the fracture strain. The bending strength is 236MPa at 1200?, reaches the maximum value(294MPa) at 1400?, then decreases gradually with the increase of temperature. The bending strength was slightly higher at 1800? than that at the initial test temperature(1200?), indicating that the C/C-SiC composites still had excellent mechanical properties at ultra-high temperature. The difference of mechanical properties of C/C-SiC composites before and after 1400? is caused by the change of the physical state of residual silicon in the composites.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 1927

# Manufacturing of CMC Cf/(C-SiC)m with Improved Gas Tightness from Monomethylsilane CVI Method in a Cold Walls Reactor


Presenting author: Dr TIMOFEEV Ivan
Affiliation: JSC Kompozit
Pionerskaya, 4,141070 Korolev (Moscow reg., Russia)
Co-authors: KOLOMIYTCEV Ivan (JSC Kompozit), TIMOFEEV Anatoliy (JSC Kompozit), BOGACHEV Eugeniy (JCS Kompozit)

Timofeev I.A., Kolomiytcev I.A., Timofeev A.N., Bogachev E.A.
JSC Kompozit, 4 Pionerskaya, Korolev, Moscow region 141070, Russia



Chemical vapor deposition Si? matrix from monomethylsilane (MMS) characteristically exhibits simplicity of gaseous medium composition, relatively low process temperature and ecological cleanness of both initial reagent and reaction products.

The talk addresses the data about formation process of silicon carbide matrix into low-density axisymmetric carbon-carbon preforms under direct resistance heating in an unsteady thermal field. Carbon-carbon preforms were manufactured with overbraiding technology and carbon CVI. The chosen combination of process parameters allows to reach the density near 2.1 g/cc during 110 hours of SiC CVI. Kinetics of CMC density growth and microstructure of the ceramic matrix obtained depending from process parameters has been investigated; test results are demonstrated (including gas tightness tests data).

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 2443

# Fabrication and characterization of PIP based C/SiC composites using the indigenous SiC precursor

Presenting author: Dr KUMAR Suresh
Affiliation: DMSRDE
GT Road,Kanpur,208013 Kanpur (Uttar Pradesh, India)

Co-authors:

Multidirectional continuous fiber reinforced Carbon-Silicon Carbide (C/SiC) composites have been identified for several advanced ultrahigh temperature applications. These composites are manufactured based on polymer impregnation and pyrolysis (PIP)process using the indigenous polycarbosilane polymer precursor and the fine tuning of the fabrication methodologies is accomplished by design of experiments. The studies conducted have revealed that the fiber volume fraction, pyrolysis temperature and density are the most effective process parameters. The composites have been characterized for flexural strength, tensile strength, thermal diffusivity and coefficient of thermal expansion (CTE). The study, thus, conducted has enabled the optimization of process parameters and using these optimized process conditions, typical size test articles have been fabricated to establish the feasibility to fabricate shaped products such as nozzle and hot structures. These results are briefly described in the following sections and the same will be described in detail in the technical presentation. Uni-directionally and bi-directionally carbon fiber reinforced C/SiC composite were prepared using T300 carbon fibers. The fibrous preform was impregnated with resin solution of polycarbosilane (PCS)and di-vinyl-benzene (DVB). The impregnated preforms were consolidated/processed by heating them up to 300oC under pressure to obtain fiber volume fraction(Vf)of the order of 55%.The consolidated composites were pyrolyzed up to 1600oCand were further infiltrated with PCS resin using vacuum infiltration. Impregnation and pyrolysis steps were repeated up to six times to get composites having density up to 1.8 g/cm3. Flexural stress of the composites was determined as per ASTM C-1341 using 3-point bending fixture while tensile stress was measured using ASTM C-1273-15. Coefficient of thermal expansion of the composite was measured up to 1000oC in a dilatometer using 5x5x25mm specimens. Thermal diffusivity of the composite samples was measured up to 1200oC as per ASTM E-1461-13 standard. Flexural and tensile strength of the UD composites were found to be varied between 550-650 MPa and350-450 MPa respectively. While, for the 2D C/SiC composites, flexural and tensile strength were found to be 300-350MPa and 150-230MParespectively.The properties were greatly influenced with the process parameters such as, Vf, interface characteristics and pyrolysis temperature. The strength was highest for composites heat treated at 1400oC and exhibitedincreasing trend with increasing Vfand densification. Higher strength at 1400oC is interpreted due to the phase composition and shows extensive fiber pull out and weak interface. At higher processing temperature, the fibers react with the inherent oxygen present in the matrix and results into a relatively brittle interface and cause for strength reduction for composites pyrolyzed at 1600oC. CTE varies from 2.5x10-7to 2.2x10-6m/m oC in temperature range of 200 to 1000oC.Thermal diffusivity decreases with temperature from 32 mm2/s to 7 mm2/s in the temperature range of 25-1200oC. Details of the process and mechanism along with micro-structure would be discussed in the technical presentation. In summary, processing method for the C/SiC composites is established by fine tuning the most effective process parameters through design of experiments. Mechanical and thermal properties of these composites are found to be comparable with internationally reported values.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 2156

# Influence of machining on the surface roughness and the bending strength of 2d - fabric reinforced C/C-SiC

Presenting author: M RöSIGER Achim
Affiliation: Hochschule Augsburg, Department of Mechanical and Process Engineering
An der Hochschule 1,86161 Augsburg (Germany)

Co-authors: GOLLER Ralf (Hochschule Augsburg Department of Mechanical and Process Engineering)

Surface finishing processes are required to generate the final shape of CMC (Ceramic Matrix Composite) components. Diamond grinding is still the most effective machining method for manufacturing C/C-SiC components like ceramic brake discs. Turbine vanes, shrouds and combustion parts are new applications which have 3d shapes and designs. Therefore the milling process with polycrystalline diamond tools becomes an interesting option for more economic and reliable production.
Machining involves high specific loading, which is concentrated in the contact zone between tool and work piece. CMCs show typical brittle material removal mechanisms, which lead to micro fragmentation on the surface and micro cracking in the subsurface.
In this study grinding and milling tools are compared and used to machine a 2d fabric reinforced C/C-SiC with different speed and efficiency. During machining the applied loads were measured by a piezoelectric 3d sensor. The influence of the different machining conditions is evaluated regarding the surface quality (cracks, roughness) and the strength. The roughness of the machined surfaces was measured by optical focus variation method and evaluated according DIN EN ISO 25178. For the strength investigation bending specimens were prepared and tested according to DIN EN ISO 658-3.
Especially process conditions with high material removal rates show increased cutting loads together with high surface roughness.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 1984

# Design of a pilot-scale Microwave Heated Chemical Vapor Infiltration plant: An innovative approach


Presenting author: M D'AMBROSIO Roberto

Affiliation: Department of Civil and Industrial Engineering, University of Pisa
Via Diotisalvi, 2,56122 Pisa (Italy, Italy)

Co-authors: ANNINO Giuseppe (Istituto per i Processi Chimico-Fisici IPCF-CNR), CINTIO Andrea (Istituto per i Processi Chimico-Fisici IPCF-CNR), LAZZERI Andrea (Department of Civil and Industrial Engineering University of Pisa)



A hybrid Microwave assisted Chemical Vapor Infiltration (MW-CVI) pilot plant to produce silicon carbide-based ceramic matrix composites (SiCf/SiC) was designed, built and setup, as a part of the European project HELM. Different from existing lab-scale MW-CVI equipment, the design of this pilot plant was carried out with the idea of a further industrial scale-up, avoiding any lab scale solution.

In order to tailor a suitable temperature profile in the preform to be infiltrated, this pilot plant was designed to use a combination of conventional and microwave heating. In particular, the inner chamber of the reactor and all the components exposed to high temperature and extreme chemical environment were built in high-quality graphite, whose microwave conductivity was experimentally determined. Due to the complexity of the procedure required for a proper heating of large samples, the reactor was designed with the internal microwave cavity acting as an overmoded resonator at the frequencies of interest. The electromagnetic behavior of the resonant cavity, both empty and loaded with sample and sample holder, were accurately investigated by means of rigorous numerical modelling based on Comsol Multiphysics software. The numerical modelling was extended to include the microwave heating of the sample, both in the static case and for rotating samples, including the dielectric and thermal properties of the sample as a function of the temperature, which were experimentally determined within the project HELM. The study of the combined electromagnetic and thermal problem enabled the determination of the temperature homogeneity in realistic working conditions, as well as the microwave power necessary to reach the desired temperature.

The main steps of the design, as well as the results of the first infiltration tests showing the necessary reverse temperature profile in the samples, will be discussed in this contribution, together with the obtained reaction efficiency.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 1882

# Enhanced Mechanical and thermal conductivity of Carbon fibre-reinforced silicon carbid by Laser-Machining-Assisted CVI


Presenting author: Dr WANG Jing
Affiliation: Northwestern Polytechnical University
NPU P.O.Box 547,West Youyi Road 127,710072 Xi'an (Shaanxi, China)

Co-authors:



Carbon fibre-reinforced silicon carbide (C/SiC) have received much attention in the aircraft, aerospace, et al. due to their low density, high specific strength, excellent mechanical properties and good electromagnetic (EM) wave absorbing properties. A novel technique based on laser-machining-assisted chemical vapor infiltration (LA-CVI) was proposed and developed to fabricate C/SiC. During LA-CVI, micro-holes was machined by laser, which performing as infiltration-assisting holes for reactant gas. The results showed that the bending strength of the C/SiC composites increased by 12%, respectively, compared with those of classical CVI-C/SiC composites. Analyzed by using scanning electron microscopy, LA-CVI C/SiC composites displayed significantly improved damage-tolerant fracture behavior. Moreover, after filling the channel with SiCw, the bending strength of the C/SiC composites increased by 25%. And the thermal conductivity had improved greatly, about 4 times of C/SiC composites. The advantages of the LA-CVI method with regard to improving mechanical properties and the thermal conductivity of C/SiC were demonstrated.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 2181

# Tailored macro-pores during the formation of C/C-SiC via liquid phase pyrolysis

Presenting author: Dr FLAUDER Stefan
Affiliation: University of Bayreuth, Department of Ceramic Materials Engineering
Ludwig-Thoma-Straße 36b,95447 Bayreuth (Germany)

Co-authors: LANGHOF Nico (University of Bayreuth Department of Ceramic Materials Engineering), KRENKEL Walter (University of Bayreuth Department of Ceramic Materials Engineering)

The synthesis of C/C-SiC via liquid silicon infiltration (LSI) with thermoplastic carbon precursors can lead to the formation of interims macro-pores in the C/C state. The macro-pore pattern formation can be controlled and used as a new level in microstructure to derive new kinds of structural and functional ceramic matrix composites. The pore formation occurs in the CFRP state with the re-melting of the thermoplastic matrix and can be fixed to the C/C state after liquid phase pyrolysis. The macro-pores are primarily induced by a non-linear elastic recovery of the fiber preform and not via gas formation and release during pyrolysis. The formation of the macro-porosity, macro pore shape and macro pore size are described and explained. These pores can be tailored by a precise process control in size and shape. The pore shapes can vary from isolated spherical pores to an interconnected tube like macro-pore network. The isolated pores can be adjusted in the size of a few 10 µm up to a few 100 µm. The cross sections of the channels of the tube like pore network can be set in the range of some 10 µm to a few 100 µm. The relationship between starting setting variables of the fiber preform, the process conditions, and the resulting structure are discussed. The prospects and limits of the structure control via intrinsic pore formation are discussed with regard to the starting fiber volume fraction and the driving force of the pore formation. The detailed mechanism of the macro-pore formation are presented and the potential of the process for new CMC materials is discussed.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 1975

# SiC-C slurries for a colloidal manufacturing of SiC/SiC composites


Presenting author: M HELD Alexander

Affiliation: University of Bayreuth
Ludwig-Thoma-Straße 36b,95447 Bayreuth (Bavaria, Germany)

Co-authors: RANK P. (Department of Ceramic Materials Engineering University of Bayreuth Germany), PUCHAS G. (Department of Ceramic Materials Engineering University of Bayreuth Germany), KRENKEL W. (Department of Ceramic Materials Engineering University of Bayreuth Germany)



The demand of materials with an outstanding thermo-mechanical performance and low density for applications in aeronautics, space and gas turbines is increasing. For this purpose, SiC-based ceramic matrix composites (CMCs) are superior compared to conventional Ni-based superalloys.

A colloidal SiC/SiC manufacturing route based on SiC-C slurries is described. This facilitates a faster and more economical way to produce these non-oxide CMCs, avoiding the costly and time-consuming pyrolysis or gas deposition steps of conventional SiC/SiC routes. The manufacturing process is subdivided into three steps, starting with a fiber coating, followed by the slurry impregnation of the fiber preform, and the infiltration with molten silicon to obtain a dense and homogeneous SiC/SiC composite. The development of aqueous SiC-C slurries is described and the influence of different raw materials on viscosity, drying behavior and residual silicon are addressed. The effect of varying carbon sources on the slurry, the green body and the resulting ceramic will be discussed in detail.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 2213

# SiC fiber-reinforced CoSi matrix composite fabricated by melt-infiltration processing


Presenting author: Dr AOKI Takuya

Affiliation: Japan Aerospace Exploration Agency (JAXA)
6-13-1 Osawa,1810015 Mitaka (Tokyo, Japan)

Co-authors: TSUNOURA Toru (Tokyo Institute of Technology), YOSHIDA Katsumi (Tokyo Institute of Technology), HAYAMA Naoki (Tokyo University of Science), TAKAYA Sawahiraki (Tokyo University of Science), RYUTA Kitamura (Tokyo University of Science), SHINJI Ogihara (Tokyo University of Science), TOSHIO Ogasawara (Tokyo University of Agriculture and Technology)



Silicon melt infiltration (Si-MI) is one of the practical processing employed for the matrix formation of SiC fiber-reinforced SiC-based matrix composites (SiCf/SiCs). Principally, free Si remains in the matrix after Si-MI processing and limit the heat resistance of the composite bellow the melting point of the Si (1414 ?C). In this study, in order to improve the heat resistance, cobalt monosilicide (CoSi) matrix was formed by a MI processing, because CoSi shows a higher melting point (1455?C) than pure Si.

The MI processing was carried out at 1500?C under vacuum using a graphite furnace. X-ray CT inspections revealed that the porosities remained in the fiber preform after CVI-SiC treatment were successfully filled with CoSi matrix. The open porosity after the MI processing was less than 5 vol.%.

Three-point bending tests were carried out up to the temperature of 1400?C in an inert atmosphere. The SiCf/CoSi composite prepared in this study maintained high stiffness and strength up to 1400?C. Whereas, the composite melt-infiltrated with pure Si degraded significantly at 1400?C. Moreover, a part of Si matrix came out from the specimen due to melting during the bending test at 1400?C.

In the presentation, oxidation test results of the SiCf/CoSi composite in a steam environment will be also introduced.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 1998

# Evaluation of property and performance of SiCf/SiC composites


Presenting author: Dr PARK Ji Yeon

Affiliation: Korea Atomic Energy Research Institute
111, DAEDEOK-DAERO 989BEON-GIL,YUSEONG-GU,34057 DAEJEON (South Korea)

Co-authors: KIM Daejong (Korea Atomic Energy Research Institute), LEE Hyeon Geun (Korea Atomic Energy Research Institute), KIM Weon-Ju (Korea Atomic Energy Research Institute)



SiC ceramics are known to have excellent properties in terms of their thermal resistance, oxidation resistance, high strength, and chemical stability at higher temperatures. Therefore, it is a promising material for structural applications at high temperature. In this study, a tubular composite was prepared by chemical vapor infiltration method to evaluate the mechanical properties of SiCf/SiC and a plate composite was prepared by electrophoretic deposition (EPD) to observe the oxidation behavior. Tubular preforms were manufactured with filament winding method using Tyranno-SA3TM SiC fibers. A thin pyrolytic carbon (PyC) layer (~200 nm) was deposited onto the SiC preform fibers. Matrix filling was performed using methyltrichlorosilane (MTS). The matrix filling behaviors of the CVI-SiCf/SiC tubes were investigated with the different thicknesses and the diameters. The C-ring compression strength was measured with the different widths of the specimen. To produce the plate composite for oxidation testing, the two-dimensional plain woven SiC fabric (Tyranno SA3TM) was coated by PyC followed by an additional coating of SiC to prevent any reaction with the sintering aid that could occur during hot pressing. The matrix was filled with commercial ?-SiC powder (4620 KE, Nano Amor Inc., USA) with 12 wt.% Al2O3-Y2O3 sintering additive by AC- EPD. Twenty layers of SiC fabric with ?-SiC powder embedded in the matrix using EPD were stacked to carry out the hot pressing at 1750oC and 20 MPa for 2 h under Ar atmosphere. The oxidation behaviors of EPD SiCf/SiC were evaluated by ablation test. The ablation temperature range is 1300oC ~ 2000oC and the oxidation time is 30 min. To investigate the oxidation behaviors, weight change measurement, microstructure observation, and phase analysis were conducted.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 1850

# The preparation, microstructure and mechanical properties of SiC chopped fiber reinforced quasi-isotropic SiC-based composites


Presenting author: M XU Zeshui
Affiliation: Northwestern Polytechnical University
No. 127, Youyi Road(West),710072 Xi'an (Shaanxi, China)

Co-authors: CHENG Laifei (Northwestern Polytechnical University), YE Fang (Northwestern Polytechnical University)



Continuous fiber-reinforced SiC ceramic composites (CFCC-SiC) have low density, high mechanical properties, good oxidation resistance and non-catastrophic failure. However, the structure of the fiber preforms (such as 2D, 2.5D and 3D) results in the anisotropy of the CFCC-SiC. In this paper, for the first time, quasi-isotropic SiC chopped fiber preforms were manufactured via air-laid process in order to improve the anisotropy of the CFCC-SiC. The randomly distributed SiC chopped fiber in preforms had a volume fraction of 6.9 % so that the preforms with the high porosity and large pore size could be subsequently densified by Chemical Vapor Infiltration (CVI) process. The microstructure, the phase composition, the pore-size distribution and the mechanical behavior of the SiC chopped fiber reinforced SiC ceramic composites were investigated. The bulk density and open porosity of the as-prepared SiCf/SiC with low fiber volume fraction were 2.45 g/cm3 and 19.0 %, respectively. Moreover, the flexural strength and the interlaminar shear strength were severally 122.23 ± 11.76 MPa and 68.28 ± 10.66 MPa. At the meantime, the preforms and the matrix of the carbon chopped fiber reinforced SiC ceramic composites were prepared by the same process so as to compare with the quasi-isotropic SiCf/SiC.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 2094

# Tungsten fibre-reinforced tungsten composite – extrinsic toughening of a new high performance material

Presenting author: Dr RIESCH Johann
Affiliation: Max-Planck-Insitut für Plasmaphysik
Boltzmannstraße 2,85748 Garching bei München (Bavaria, Germany)

Co-authors: BALDEN Martin (Max-Planck-Institut für Plasmaphysik), CURZADD Bailey (Max-Planck-Institut für Plasmaphysik), GIETL Hanns (Max-Planck-Institut für Plasmaphysik), GREUNER Henri (Max-Planck-Institut für Plasmaphysik), HöSCHEN Till (Max-Planck-Institut für Plasmaphysik), KäRCHER Annemarie (Max-Planck-Institut für Plasmaphysik), LINSMEIER Christian (Forschungszentrum Jülich GmbH Institut für Energie- und Klimaforschung – Plasmaphysik), MAO Yiran (Forschungszentrum Jülich GmbH Institut für Energie- und Klimaforschung – Plasmaphysik), RAUMANN Leonard (Forschungszentrum Jülich GmbH Institut für Energie- und Klimaforschung – Plasmaphysik), SCHWALENBERG Daniel (Forschungszentrum Jülich GmbH Institut für Energie- und Klimaforschung – Plasmaphysik), YOU Jeong-Ha (Max-Planck-Institut für Plasmaphysik), COENEN Jan (Forschungszentrum Jülich GmbH Institut für Energie- und Klimaforschung)

The development of capable materials is essential for sophisticated future energy systems, especially for high-heat-flux-loaded areas in a future fusion power plant. Due to a unique property combination, tungsten (W) would be the ideal material for this application. However, tungsten is brittle at room temperature and prone to operational embrittlement. Tungsten fibre-reinforced tungsten composites (Wf/W) utilize extrinsic mechanisms to improve the fracture toughness and thus mitigate the brittleness of W in a manner similar to ceramic fibre-reinforced ceramic composites [1].
In this contribution, we will present the main characteristics and properties of this new composite material. Drawn potassium-doped W wire is used as ductile, high strength fibres and engineered fibre-matrix interfaces ensure the desired composite behaviour. The matrix is formed around a woven long-fibre or randomly orientated short-fibre preform either by the chemical deposition of tungsten (CVD) or by a powder metallurgical process. A review will be given of the material's development history, starting with the first ideas and proof of principle up to operational demonstration where the material is tested at high heat fluxes of up to 20 MW/m2. Highlights are the development of a continuous manufacturing routine for CVD and mechanical characterisation with in situ synchrotron tomography.
As the matrix material in Wf/W is brittle, the composite behaviour is very similar to fibre-reinforced ceramics. A comparison of the two composite groups with emphasis on toughening

mechanisms, manufacturing, and characterization techniques will point to common problems and possible trade-offs. Here, the focus will be on fracture mechanical characterisation and the contribution of the W fibre's ductility to the toughening behaviour. Finally, we discuss further development needs and next step projects leading to the use as a fusion reactor material, as well as possible future applications beyond fusion for both Wf/W and W fibres.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 2024

# THE DIAMOND-CARBID-SILICON COMPOSITE "SKELETON": STRUCTURE, PROPERTIES, USE PERSPECTIVES IN AVIATION PROPUL-SION


Presenting author: M MEZENTSEV Mikhail
Affiliation: Central Institute of aviation motors by name P.I. Baranov
Aviamotornaya 2,111116 Moscow (Moscow, Russia)

Co-authors: KARIMBAEV Telman (Central Insitute of Aviation Motors by name P.I. Baranov), GORDEEV Sergey (Central Research Institute of Materials)



Analysis of properties of sic ceramic and diamond shows that their combination should lead to composites with a unique combination of stiffness, conductivity, hardness, and wear-resistance. Such materials can effectively be treated as construction materials.
The diamond carbid-silicon composites "Skeleton" (DCS "Skeleton") is obtained by the formation of preform from diamond powders with the subsequent synthesis of silicon carbide manufactured parts. This technology has been successfully tested on products of a fairly large size and complex forms, providing unshrinkable final product at moving from preform to one.
The DCS "Skeleton" structure is without porous and consists of three phases-diamond, silicon carbide and silicon. The relation of this phase can be changed by technological tech-niques [1].
The "Skeleton" materials have high rigidity: modulus of elasticity may reach a record values-800 HPa (isotropic material). Specific stiffness-ratio of composites elastic modulus and density on 15-35% higher than that of beryllium and SIC ceramic. Strength of composites is 250-350 MPa. Heat resistance and strength at high temperature of composite is provided by high thermal conductivity (300-450 w/m * k) and low CTE (2 * 10-6 K-1) [1].
In CRIM2 jointly with specialists of the CIAM1 have developed various composite de-sign of nozzle apparatus (Fig.1) from DCS "Skeleton", having blocks with separate shelves and blades, bloks with monolithic shelves and blades, and monolithic blocks with shelves and two or more blades allow the use of DCS "Skeleton" in uncooled parts and nodes of aircraft engines with operating temperature up to 1550°C.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 1987

# Thermosetting injection moulding for shaping of C/C-SiC-ceramics: Influence of flow direction and weld lines


Presenting author: M STILLER Jonas

Affiliation: Chemnitz University of Technology - Institute of Lightweight Structures
Reichenhainer Straße 31-33,09126 Chemnitz (Germany)

Co-authors: NESTLER Daisy (Chemnitz University of Technology - Institute of Lightweight Structures), PäßLER Erik (Chemnitz University of Technology - Institute of Lightweight Structures), KEMPE Fabian (Department of Polymer Chemistry), AHMAD Husam (Institute of Material Science and Engineering), KROLL Lothar (Chemnitz University of Technology - Institute of Lightweight Structures), SOMMER Michael (Department of Polymer Chemistry), WAGNER Guntram (Institute of Material Science and Engineering)



Ceramic-matrix composites (CMC) made of carbon and silicon carbide dual matrix reinforced with carbon fibres (C/C-SiC) have exceptional heat, thermal shock, creep, and wear resistance, while also having low density and high strength. In comparison to monolithic ceramics, CMC possess ductility and damage tolerance, which opens this material for severe applications. Starting in space applications, this material is today well established in friction applications, where lightweight high-performance brakes securely decelerate e.g. luxury cars or elevators. The high production costs still limit the broad application like as brake discs in standard passenger cars, although less weight, better performance and longer lifetime. The industrial used production process is the liquid silicon infiltration (LSI) with it three steps: green body shaping, pyrolysis and silicon infiltration.

In this work, the shaping process of the carbon fibre reinforced plastic (CFRP) green body is done by thermoset injection moulding, which enables large-scale manufacturing. The used matrix polymers are phenol and phenylphenol resin in different shares as well as varied proportions of chopped carbon fibres as reinforcing structure are examined. Industrial equipment for compounding and injection moulding processes the ingredients and demonstrator-scale CFRP-green bodies are the outcome. Test specimens are cut out of the demonstrator in different directions to investigate influences of flow direction, weld lines and tool geometry. The samples pass the LSI process (pyrolysis and silicon infiltration) and their microstructure and mechanical properties are investigated in all three manufacturing states CFRP, C/C and C/C-SiC. Analysis of the porosities, densities and shrinkages in the three states complete the work. All outputs are compared with previous results and to the state of the art.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 1856

# Influence of the pyrolysis process parameters in the production of short fibre-reinforced C/C-SiC composites

Presenting author: Mme NIER Natalia
Affiliation: Chemnitz University of Technology
Erfenschlager Strasse 73,09125 Chemnitz (Saxony, Germany)

Co-authors: AHMAD Husam (Chemnitz University of Technology), GURK Hannes PäßLER Erik RODER Kristina NESTLER Daisy KROLL Lothar WAGNER Guntra

A new short fibre-reinforced C/C-SiC composite is developed by using the liquid silicon infiltration (LSI) process, which consists of three processing steps. At first, a carbon fibre reinforced plastic (CFRP) composite is fabricated. The shaping of this composite is realised by an injection moulding process. Afterwards the CFRP composite is converted in a porous C/C composite by pyrolysis. In the third step, the Liquid silicon is infiltrated to form a dense C/C-SiC composite.
One of the most important aspects in the LSI route is the porosity in the C/C state. Due to the fact, that the innovative manufacturing process requires other starting materials, the porosity is influenced and must be adapted. One possibility method is pyrolysis. In this paper the influence of the pyrolysis, parameters are examined. The microstructures of the composites (CFRP, C/C and C/C-SiC composites) are characterised. These investigations show a relationship between the used parameters of pyrolysis process and the forming of the porosity and the properties of the finished C/C-SiC composites. In regards to the optimisation of the process, an optimal process condition is specified.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 1949

# Characterization of SiC slurry impregnated SiC/SiC composite fabricated by LSI process


Presenting author: Dr SEONG Young-Hoon

Affiliation: Korea Institute of Energy Research
152 Gajeong-ro,Yuseong-gu,34129 Daejeon (South Korea)

Co-authors: CHOI Jaehyung (Korea Institute of Energy Research), KIM Seyoung (Korea Institute of Energy Research), KIM Soo-hyun (Korea Institute of Energy Research), HAN In-sub (Korea Institute of Energy Research), WOO Sang Kuk (Korea Institute of Energy Research), BANG Hyung Joon (Korea Institute of Energy Research)



Materials and cooling technology are the most important core technologies for satisfying the required lifetime and stable operation of components that require high temperature for high output / high efficiency in gas turbine. In order to apply metals to H-Class, that is, 1600?, considering that the temperature limit of the super-alloy is 950?, even though it is possible to lower the temperature by 150? with thermal barrier coating, it is necessary to lower the temperature by more than 500? using cooling technique. This results in a significant reduction in the power output and efficiency of the gas turbine. Development and demonstration of CMC material are needed as the only alternative to solving turbine part material technology and cooling technology problem caused by high temperature of TIT (Turbne Inlet Teperature). In order to improve the physical properties of CMC materials, the properties of fibers and matrix should be secured together. Specifically, fiber protection coating technology, high strength matrix manufacturing technology, and composite densification technology should be secured. In this study, we have studied the ceramic filler based slurry for high strength matrix using liquid silicon infiltration (LSI) method. SiC slurry was applied to suppress residual silicon and increase strength, and material properties were evaluated after the LSI process according to the composition of slurry. The SiC/SiC composites prepared by SiC slurry impregnation method and LSI method were found to have improved mechanical properties at high temperatures.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 2128

# Microstructure observation and high-temperature stability analysis of C/SiOC composites


Presenting author: Prof WANG Zhen
Affiliation: Shanghai Institute of Ceramics Chinese Academy of Science
Street (line 1)Dingxi Rd. 1295,Street (line 2),200050 Shanghai (Shanghai, China)

Co-authors: WU Qingqing (Shanghai Institute of Ceramics Chinese Academy of Science), DONG Shaoming (Shanghai Institute of Ceramics Chinese Academy of Science), YANG Jinshan (Shanghai Institute of Ceramics Chinese Academy of Science)



Cf/SiOC composites fabricated through a modified PIP process using molten MK resin as precuesor. The as-fabricated composites via 8 cycles of PIP possessed a three-point-bending stress of (V_porosity=6.2%, ?=1.82g/cm3) and excellent mechanical properties with bending strength of 312±25MPa and show obvious non-brittle damage behavior. The microstructure evolution accompanied in densification and heat-treatment processes was observed by micro-CT and SEM. The composition changes as well as strength variations during heat-treatment process were studied.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 2133

# Effect of material composition and additives on the properties of C/C-SiC composites produced via the transfer/injection moulding and liquid silicon infiltration route


Presenting author: M AHMAD Husam

Affiliation: Chemnitz University of Technology, Chair of Composites and Material Compounds
Thüringer Weg 32,09126 Chemnitz (Germany)

Co-authors: STILLER Jonas PäßLER Erik KEMPE Fabian NESTLER Daisy SOMMER Michael WAGNER Guntram KROLL Lotha



The properties of CFRP green bodies, determined by the used initial constituents such as fibre content and the matrix composition, affect significantly the physical, mechanical and micro-structural properties of the C/C-composites after the pyrolysis process. Consequently, C/C intermediate preforms with different crack-patterns and open porosities are generated. This has a profound impact on the subsequent infiltration and final properties of the resulted C/C-SiC composites. For this purpose, compounds on the basis of Novo-lac/Hexamethylenetetramine with different chopped fibre contents and additives such as Phe-nylphenol resin, silicon, and silicon carbide powders are produced utilizing a laboratory tem-pered sigma-blade kneader. The manufacturing of CFRP green bodies is then performed using a mini transfer moulding machine. One of the key points of this contribution represents the in-situ siliconisation through the co-mixing of silicon powder during the compound production and the comparison with the external silicon infiltration process. To clarify the influence of the siliconisation type and different material variations, investigations of several material proper-ties such as density, open porosity, shrinkage behaviour, bending strength, phase compositions and microstructure during different production steps are conducted. Based on the results gained from the transfer moulding technique, the feasibility of selected material compositions through the injection moulding technology is tested.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 1967

# Factorial Design of Experiments for Electrophoretically Deposited Multi-Layered Fibre-Matrix Interphase Coatings


Presenting author: M ROBERTSON Taylor
Affiliation: National Research Council of Canada
1200 Montreal Road,Building M13,K1A 0R6 Ottawa (Ontario, Canada)

Co-authors: KEARSEY Rick (National Research Council of Canada), HUANG Xiao (Carleton University)



Ceramic matrix composites (CMC) are projected to replace metallic superalloys in applications where the benefits of higher temperature capability outweigh the high costs associated with CMC production and certification. One of the main reasons for the added overall cost is the application of the fibre-matrix interphase to the ceramic fibres during manufacturing of CMC. The fibre-matrix interphase is typically produced through a chemical vapor deposition (CVD) method which has the advantages of being a non-line of sight method capable of producing thin films. The drawbacks of CVD include the high cost of the equipment and limited coating compositions. Electrophoretic deposition of fibre-matrix interface coatings has the potential to reduce the cost of producing fibre-matrix interphases while at the same to incorporate a degree of environmental barrier function into the fibre-matrix interphase itself. This work uses a factorial design of experiments method to optimize the electrophoretic deposition technique used to process multilayered fibre matrix interphase coatings. In this work a total of seven variables are examined: fibre type (four levels), coating type (four levels), electric field strength (three levels), solvent pH (three levels), coating time (three levels), dispersant (two levels), and binder (two levels). Mini-composites consisting of a central fibre bundle, electrophoretically deposited fibre-matrix interphase and a thin matrix coating produced through reactive melt infiltration were fabricated. Fibre matrix interphase properties were determined through stress-strain cycling of the mini-composites above their proportional limit. Post processing of the stress-strain hysteresis curve was used to determine the fiber-matrix interphase's debond energy and frictional sliding resistance. Metallographic examination of the samples prior to the matrix production and post fracture has also been performed and reported in this paper. Future work is targeted at integrating the electrophoretic deposition process into prepreg fabrication in order to streamline the processing of these ceramic matrix composites.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 2616

# LIGHTWEIGHT CERAMIC MATRIX COMPOSITES OF B4C INFILTRATED BY AL-ALLOY

Presenting author: Mme FARIA Mónica Filipa
Affiliation: University of Aveiro- Ceramic and Materials Engineering Dept.
Campus Universitário de Santiago,3810-183 Aveiro (GUIMARÃES, Portugal)

Co-authors: OLIVEIRA Nuno (Dept. Materials Eng. & Ceramic CICECO), GIRãO Violeta (Dept. Materials Eng. & Ceramic CICECO), OLIVEIRA F.J (Dept. Materials Eng. & Ceramic CICECO), VIEIRA J.M (Dept. Materials Eng. & Ceramic CICECO)

The development of lightweight ceramic materials is under continuous scrutiny by the manufacturers and end users. Research on advanced ceramics is equally important in the study of system components resistant to projectile impacts. Different ceramics have been used in this field, mostly alumina-based ceramics and/or non-oxide ceramics (B4C, Si3N4, AlN, amongst others). These materials have good thermal stability, high Young's modulus, extreme hardness and good abrasion resistance.

In this work, we present an alternative to obtain a final hard ceramic, with a remarkable densification and low density: ceramic matrix composites (B4C-Al) prepared by pressureless infiltration.

B4C samples with different compositions of coarse-medium-fine (C-M-F) powders were homogenized using ball milling in water and pressed into porous preforms. These preforms were then infiltrated by a molten Al-alloy, under vacuum, with infiltration temperature varying between 1250 and 1350 °C.

Samples with 70 vol.% of coarse powders and 30 vol.% of fine powders (70C-30F) achieved a green densification of 67.5%. However, the infiltration was compromised.

On the other hand , samples with 45 vol.% coarse, 45 vol.% medium and 10 vol.% fine powder (45C-45M-10F) of B4C samples reached a densification of 64.5 % and the infiltration at 1300 °C resulted in a fully densified material where the absence of pores is confirmed by microstructural analysis. This result indicates that the particle's size distribution of the preforms powders play a key role on the green density of the pieces and, consequently, on the final density and hardness of the composites

The 45C-45M-10F samples tend to have a considerable hardness (13.6 GPa) and low density (2,63 g.cm-3). XRD results show that boron carbide, boron silicon carbide and aluminum silicon carbide are the major crystalline phases formed in the composites.

The influence of the added amount of molten alloy and infiltration temperature are discussed in this work.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 2225

# Damage accumulative behavior of short carbon fiber reinforced silicon carbide under compression


Presenting author: M TOBATA Yuta

Affiliation: Graduated University for Advanced Studies
3-1-1 Yoshinodai, Chuo-ku,252-5210 Sagamihara (Kanagawa, Japan)

Co-authors: GOTO Ken (Japan Aerospace Exploration Agency), TAKEUCHI Shinsuke (Japan Aerospace Exploration Agency)



In this study, compressive test was conducted to investigate affection of damage accumulation on mechanical properties of short carbon fiber reinforced silicon carbide composite fabricated by Si infiltration. Before fracture of specimen, stress-strain relationships were measured at each peak stress to investigate permanent strain and compressive modulus. In addition, crack growth process was observed to measure crack propagation direction angle and length. Crack observation showed that cracks mostly propagate in fiber bundle orthogonally oriented to compressive direction and stopped when it reached interface between fiber bundles. Measurement of crack propagation direction angle showed that many of cracks propagated parallel to loading direction. From these results, governing damage factor seems to be mode 1 propagation in fiber bundle orthogonally oriented to compressive direction. Stress-strain relationships showed increase of permanent strain and decrease of loading modulus. However, unloading modulus didn't change at all. Thus, it seems that crack propagation in fiber bundle orthogonally oriented to compressive direction didn't affect decrease of composite compressive modulus. Considering the above results, damage accumulation behavior is concluded that cracks in fiber bundle orthogonally oriented to compressive direction propagated with mode 1 and make increase of permanent strain while compressive modulus didn't change until total fracture of composite.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 1923

# Investigation on secondary heat treatment of CVI densified ceramic matrix composites


Presenting author: Dr KRISHNAN Sreejith

Affiliation: VIkram Sarabhai Space Centre
Thiruvananthapuram,695022 Thiruvananthapuram (Keralam, India)

Co-authors: NAITHANI Neeraj (Vikram Sarabhai Space Centre), SREEKUMAR Ashish (Sree Chitra Thirunal College of Engineering Thiruvananthapuram)



Ceramic Matrix Composites (CMCs) areattractive candidate materials in the aerospace industry as thermos-structural elements in re-entry and hypersonic space vehicles due to their low density and higher temperature capabilities. Proper understanding of the mechanical behavior of CMCs is required for design of the hardware and simulation studies. Among the various factors affecting the mechanical behavior of CMCs, thermal residual stress (TRS) is a significant factor. TRS develops during the fabrication of CMCs and affects their mechanical behavior mainly by inducing matric micro-cracks. Alleviation of TRS becomes an important aspect for long duration space applications. It is postulated that secondary heat treatment (SHT) can affect the matrix, fiber and interphase properties, locally, and the mechanical response characteristics of the CMC, globally. The present paper presents the initial results from the study on the effect of SHT on the above aspects of a CMC.

C/PyC/SiC minicomposites were prepared using isothermal CVI process. Minicomposite approach was used in the current investigation to eliminate the fiber architecture effects on the mechanical properties and also to reduce the processing duration. SHT of the CVI densified minicomposites were carried out under two methods: conventional and microwave heating. The study showed that CVI deposited ?-SiC matrix undergoes further crystallization upon SHT. SHT has effectively brought down the thermal residual stress in the minicomposites from 108.9 Mpa to 49.8 MPa. Among the two methods, microwave SHT has shown better TRS reduction (49.8 Mpa) compared to conventional SHT (97.5 MPa). This effect has resulted in delayed matrix crack initiation during tensile testing. Ultimate tensile strength also increased from 110.2 Mpa to 141.1 MPa (conventional SHT) and 196.2 MPa (microwave SHT).

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 1916

# Fabrication of C/C-SiC composites by using high-char-yield resin

Presenting author: M YANG Jinhua
Affiliation: AECC Beijing Institute of Aeronautical Materials
Haidianqu wenquanzhen huanshancun,100095 Beijing (Beijing, China)
Co-authors: JIAO Jia

C/C-SiC composites was fabricated by using high-char-yield phenolic resin, with the char yield above 75%. The bulk density of the C/C-SiC composites was about 1.7 g/cm3. The fracture strength was 186±23MPa, and the tensile strength was 50±6MPa. The fracture behavior of the composites showed non-brittle fracture.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 2140

# High temperature dielectric characterization of SiC-based Ceramic Matrix Composites

Presenting author: Dr CINTIO Andrea
Affiliation: Istituto per i Processi Chimico-Fisici, Consiglio Nazionale delle Ricerche (IPCF-CNR)
via G. Moruzzi I,56124 Pisa (Toscana, Italy)
Co-authors: D'AMBROSIO Roberto (Department of Civil and Industrial Engineering University of Pisa), LAZZERI Andrea (Department of Civil and Industrial Engineering University of Pisa), ANNINO Giuseppe (Istituto per i Processi Chimico-Fisici Consiglio Nazionale delle Ricerche (IPCF-CNR)

In microwave-assisted chemical processes, a key quantity is given by the dielectric permittivity eps''-ieps'' of the materials to process, since it characterizes the energy absorption from the electromagnetic field. The knowledge of eps at the reaction temperatures is an essential step in the design of efficient microwave reactors.
A setup for the dielectric characterization of samples at frequency around 2.45 GHz and temperature T up to 1200 °C was built in the framework of the European project HELM (http://www.helm-project.eu/), following the cavity perturbation approach described in Hutcheon R. et al, JMPEE vol. 27, 93 (1992).
In this contribution, some relevant results concerning SiC-based samples are described. First, results are shown about SiC preform infiltrated with SiC (SiCf/SiC), for various temperatures and infiltration levels. In the investigated SiCf/SiC, a strong (superlinear) increase of eps' with the density is observed. Moreover, eps' increases monotonously with temperature. A similar behavior is observed with eps'', at least for relatively low densities.
Following, results concerning granular alfa and beta SiC samples with grains of different average size are shown. In this case the measurement method provides an average value of dielectric permittivity <eps> that takes into account the interstitial regions between grains. The ?-SiC samples show a relatively constant <eps'(T)> and a relatively low <eps''(T)>, decreasing with T. The grain size does not significantly affect the value of <eps>, with the exception of an increase of the dielectric losses with T. On the contrary, the ? –SiC sample exhibits comparatively higher values of eps' and eps'', both of which increasing with T.
The obtained results show that SiC-based ceramic materials display very different behaviors of eps(T), which depend on the density, the shape, and the crystalline structure. The observed variability in eps(T) confirms the importance of a dielectric characterization at the T of interest.

Topic: 5 - Innovative Design, Advanced Processing, and Manufacturing Technologies
processing in non-oxide composites : SiC/SiC, C/SiC, hybrid CMCs (incl short fibers)
Abstract no. 2866

# Preparation of Core-Shell powders by a fluidized-bed CVD process for ceramic matrix manufacturing


Presenting author: M EL MANSOURI Amine
Affiliation: University of Bordeaux, LCTS
3, Allée de la Boétie,33600 Pessac (France)
Co-authors: VIGNOLES G. (LCTS), BERTRAND N. (LCTS), GUETTE A. (LCTS)



Based on their high-temperature properties, SiC- matrix composites appear to be the materials of choice for replacing the heavy and low-melting point superalloys in the hot structure of jet engines. They can be produced by a variety of processing techniques, including the highly flexible chemical vapor infiltration process (CVI). Liquid silicon infiltration and slurry infiltration/hot pressing are less chemically flexible, but yield materials of lower residual porosity and higher thermal conductivity than the CVI process. The latter method can be potentially improved by using core-shell particles in the preparation of ceramic slurries. The idea of modifying SiC particles properties by surface treatment appears, then attractive by controlling phenomenon occurs during the infiltration of SiC fibers (containing SiC powders) with the liquid silicon (reactivity, wettability…). In that context, one of the interesting possibilities which has been tested is the fluidized bed chemical vapor deposition process (FB-CVD). Contacting the powder and the gas phase allows to coat each particle with a thin layer of a new materials, developing original surface conditions, and hence, controlled properties of us. The working method is turned to the study of i) fluidization conditions at low and high temperature for each particles used and ii) deposition conditions to obtain carbon films using the propane/nitrogen mixture. Alumina particles of Geldart's group A-B (easy-to-fluidize particles) are firstly treated in order to increase understanding of the process. Then, we worked with submicron-sized SiC powder, which are interesting for the manufacturing of ceramic matrix composite.

# Topic: 6

# Additive manufacturing of CMCs: 3D printing, laser sintering, etc.

Topic: 6 - Additive manufacturing of CMCs : 3D printing, laser sintering, etc.
Abstract no. 2372

# Additive manufacturing of ceramic matrix composites: focus on direct ink writing


Presenting author: Dr FRANCHIN Giorgia

Affiliation: University of Padova
Via Marzolo 9,35131 Padova (Italy)

Co-authors: SCANFERLA Paolo (University of Padova), MADEN Halide Selin (University of Padova), WAHL Larissa (University of Padova), COLOMBO Paolo (University of Padova)



Additive manufacturing (AM) technologies have the potential to disrupt conventional manufacturing processes, but very few systems are suited for ceramic materials; technologies employing polymers and metals are more established. The technological gap reflects on the development of AM processes for ceramic matrix composites (CMCs).
Nevertheless, the extrusion of polymer matrix composite inks with direct ink writing (DIW) showed high potential: it is possible to align short fibers and directional reinforcements along the printing direction by exploiting the shear stresses at the nozzle tip. A proper design would allow reinforcement in specific directions, generating bespoke components with complex shapes and enhanced mechanical properties.
Porous CMCs fabricated via DIW of a preceramic polymer based ink will be first presented. Preceramic polymers are already employed in polymer infiltration and pyrolysis (PIP); here, a system comprising of a polysiloxane and chopped carbon fibers was optimized. Rheological characterization led and validated our ink design for extrusion through fine (~400?m) nozzles. DIW was followed by pyrolysis in inert atmosphere, during which an amorphous SiOC phase was generated. Pyrolysis could cause extensive cracking of the matrix; the role of fibers and fillers in enhancing or preventing that will be discussed, together with the components' mechanical properties.
Geopolymer fiber composites will be provided as second example. Geopolymers are usually obtained through reaction of alumino-silicate powders in an alkaline solution. They consolidate at room temperature and can be processed via DIW, but also possess high resistance to fire and high temperature stability. Geopolymer matrix composites with short fibers were produced. As the inks consolidate with time in a continuous inorganic network, it is necessary to control their characteristics to allow for a suitable working time window. Carbon and basalt fibers were embedded in the matrix and successfully extruded; the morphology and mechanical properties of the samples will be discussed.

Topic: 6 - Additive manufacturing of CMCs : 3D printing, laser sintering, etc.
Abstract no. 2032

# 1½ -D PRINTING™: Additive Manufacturing of Filamentary Structures


Presenting author: Dr PEGNA Joseph
Affiliation: Free Form Fibers
10 Cady Hill Boulevard,12866 Saratoga Springs (NY, United States)
Co-authors: HARRISON Shay (Free Form Fibers)



This talk reports on a novel additive manufacturing technique, called '1½-D Printing™', which itself builds upon a patented process to grow large arrays of continuous ceramic fibers in parallel, called 'Fiber Laser-Printing'.
The Fiber Laser-Printer represents a novel class of additive manufacturing processes that: (1) is nearly material-agnostic, (2) is containerless, and (3) allows for fiber diameter variations. A material-agnostic tool is one that is independent of the workpiece material; an extremely rare property among manufacturing processes. In this instance, the Fiber Laser-Printer was used to produce small diameter (15-50 µm) fibers of boron, boron carbide, silicon carbide, and tungsten carbide among others. A containerless process in one in which the workpiece is self-supported, and is not in contact with a physical tool during the machining process; thus preventing material contamination. The output of the Fiber Laser Printer is a ribbon of parallel continuous filaments with adjustable diameter and composition.
1½-D Printing is a direct-write method that allows for multi-material coating patterns to be deposited in parallel onto a ribbon of parallel filaments as it is produced.  This feature of course allows the fabrication of Silicon Carbide directly with an interphase layer. More importantly, it allows for the fabrication of micro-devices embedded into the fiber; for example thermocouples. We illustrate this presentation by reporting on work conducted with US Department of Energy support toward the development of novel accident-tolerant nuclear technology.

Topic: 6 - Additive manufacturing of CMCs : 3D printing, laser sintering, etc.
Abstract no. 1997

# 3D PRINTING OF CERAMIC MATRIX COMPOSITES USING FUSED DEPOSITION MODELLING


Presenting author: M KUDISONGA Christian
Affiliation: The University of Queensland
Advanced Engineering Building #49,Staff House Rd,4072 St Lucia/ Brisbane (Queensland, Australia)
Co-authors: HEIZTMANN Michael (School of Mechanical and Mining Engineering The University of Queensland Australia)



Conventional Ceramic Matrix Composite (CMC) manufacturing methods require expensive moulds and it is often difficult to produce complex parts with high accuracy. There is an increasing demand for alternative and more flexible manufacturing methods. Additive Manufacturing (AM) allows custom designed parts to be fabricated with greater complexity and do not require the use of moulds. While there are several additive manufacturing processes, few allow the versatility to add reinforcements. So far, researchers have only reported the 3D printing of CMC with short fibres, through Laminated Object Manufacturing or powder bed processing methods. Because of the short fibre reinforcements, the mechanical properties of these parts remain limited. The use of continuous fibres would increase the overall structural strength of the printed part. Here we show a novel method for 3D printing with continuous fibres. This process combines the Fused Deposition Modelling (FDM) process with the Liquid Silicon Infiltration (LSI). This approach uses Lignin as a base material for the many benefits it brings. In addition to be cost-effective and already used to make carbon fibres, Lignin is a thermoplastic that can be cross-linked, so the printed part can endure the pyrolysis without losing its shape. Lignin has a relatively high carbon yield and has a tailorable processing temperature. However, the raw Lignin cannot be processed and needs to be chemically modified. In this work, we successfully show evidences of modification of the lignin by FTIR and DSC. The modified lignin demonstrated an attractive carbon yield of around 35%.

Topic: 6 - Additive manufacturing of CMCs : 3D printing, laser sintering, etc.
Abstract no. 2272

# Direct ink writing of polymer-derived ceramic composites

Presenting author: Prof COMPTON Brett
Affiliation: University of Tennessee
1512 Middle Drive,201 Dougherty Engineering Building,37996 Knoxville (TN, United States)
Co-authors:

Extrusion-based additive manufacturing (AM) technologies – including fused filament fabrication (FFF) and direct ink writing (DIW) – present opportunities to create composite materials and multi-material architectures that cannot be made using other AM technologies. The direct deposition nature of these processes enable the straightforward deposition of multiple materials though the use of multiple print heads, while the extrusion process preferentially aligns high aspect ratio filler materials like fibers, whiskers, and platelets. This alignment, coupled with the flexibility to choose the print path for each layer provides the ability to prescribe unique fiber arrangements within printed parts that simply does not exist with other manufacturing technologies.
This talk will focus on recent developments in DIW of polysilazane-based preceramic polymer formulations and the resulting ceramic composites. Special focus will be paid to the rheological requirements for successful printing, as well as the relationship between filler morphology, ink rheology, print parameters (i.e. nozzle size and print speed), and anisotropy in the printed part. Insights gained from recent studies on printed epoxy composites will be discussed in the context of ceramic composites. Finally, novel deposition strategies to control fiber orientation and create novel multi-material architectures will also be discussed. Current challenges and open questions will be highlighted throughout the talk.

Topic: 6 - Additive manufacturing of CMCs : 3D printing, laser sintering, etc.
Abstract no. 2142

# Manufacturing of silicon oxycarbide parts by stereolithography using cost-reduced Polymer-Derived Ceramics


Presenting author: M ARBORE Romain
Affiliation: CTTC
7 rue Soyouz,87000 Limoges (France)
Co-authors: BALLESTERO Anthony (CTTC), MAYAUDON Alexis (CTTC), BOYER Laurence (CTTC), DURAND Olivier (CTTC), DUEE Natacha (RESCOLL), DELIANE Florent (RESOLL)



One method to build complex shape and high performance materials is the use of additive manufacturing technique such as Stereolithography (SLA) because of its high accuracy. With SLA techniques, a material with photo-reactive components is exposed to a UV source that induces photopolymerization of cross-sectional patterns in stacked layers in order to get a 3D part, starting from a CAD file.

Today, the strategy to get high performance ceramics by SLA is to include particles into a photopolymerizable resin. Due to their absorbance, silicon carbide are difficult to obtain. To go around the problem, Polymer-Derived Ceramics (PDCs) route seems to be a convenient way to obtain SiC-based printed parts because it deals with a polymer instead of a loaded paste. The PDCs present the advantages to be custom tailored in liquid state and do not absorb in the UV zone enabling their photopolymerization. These inorganic polymers combine the properties of a polymer feedstock and the possibility to convert them into non-oxide ceramic materials such as carbides or nitrides by a thermal treatment. Nonetheless, these materials are still quite expensive.

Thanks to an R&D program granted by the Nouvelle Aquitaine Region, CCTC, specialist of ceramics and their processing, and RESCOLL, specialist in polymer formulation are collaborating since 2017 to develop effective and cheap printable PDCs for SLA.

This presentation will present the results of this fruitfull collaboration on the synthesis of PDCs and their processing by SLA. The presentation will focus on the PDC synthesis formulation in order to achieve the right processability (stability, rheology and reactivity). Then, it will deal with the definition of printing parameters, photopolymerization and optimal shaping resolution. In the end, the importance of thermal treatments to get a good ceramic conversion and good mechanical behaviour will be assessed.

Topic: 6 - Additive manufacturing of CMCs : 3D printing, laser sintering, etc.
Abstract no. 2043

# Macroporous SiC Ceramics by 3D-printing and chemical vapor infiltration/deposition


Presenting author: Dr CHOLLON Georges
Affiliation: LCTS-CNRS
3, allee de La Boetie,33600 PESSAC (France - DOM)

Co-authors: JACQUES Sylvain (LCTS), PIQUERO Thierry (CEA), ROCHAIS Denis (CEA), DAVID Patrick (CEA), CHOLLON Georges (LCTS)



Open porosity cellular silicon carbide-based ceramics have a great potential for uses in energy conversion, for instance as volumetric solar receivers. In spite of their tolerance to mechanical damage, structural applications at high temperature are still limited due to high production costs or not fully suitable processes or properties. The objective of this work was to investigate a route for the manufacturing of macroporous SiC ceramics, which is based on 3D printing and chemical vapor infiltration (CVI). The green ??SiC microporous structures were binder jetting 3D-printed in conditions leading to a high microporous open porosity, to promote the diffusion of gases and thus the following CVI process. The structures were reinforced successively by CVI and chemical vapor deposition (CVD) of SiC using a CH3SiCl3/H2 mixture in distinct conditions. The multiscale structure of the SiC porous specimens was examined by Scanning Electron Microscopy, X-ray tomography and mercury intrusion porosimetry. The elemental and phase content was analyzed at the microscale by electron probe micro-analysis and Raman spectroscopy respectively. The oxidation and thermal shock resistance of the porous SiC structures and smaller rod-like specimens was examined, as well as the thermal and mechanical properties. The pure and dense CVI/CVD-SiC coating was found to considerably improve the mechanical strength, oxidation resistance and thermal diffusivity of the material.

Topic: 6 - Additive manufacturing of CMCs : 3D printing, laser sintering, etc.
Abstract no. 1898

# Magics of 3D printed ceramic composites: Structural to Functional


Presenting author: Prof MEI Hui
Affiliation: Northwestern Polytechnical University
No 127 Youyi Westroad,710072 Xi'an (Shaanxi, China)

Co-authors:



3D printing is an innovative approach to build 3D objects with well-defined geometries directly from the CAD model, possessing unique advantages in geometrical shape design as well as rapid prototyping. Ultralight high strength lattice structures and high surface area complex 3D structure constructions can be simply fabricated, showing a promising prospect from structural to functional applications. The main progress is listed below: (i) Various ultra-light porous ceramic lattices fabricated by 3D printing technology, were mechanically improved by further chemical vapor infiltration and high temperature treatment. The printed ultra-light ceramics were also reinforced by whiskers and diverse fibers. (ii) 3D printing provides a feasible research scheme to achieve the facile fabrication of electromagnetic wave (EMW) absorbing ceramic metamaterials with complex chiral structures. EMW absorbing properties were improved by printing the optimized microstructural compositions and macrostructures of the ceramic metamaterials. (iii) A higher photocatalytic efficiency was obtained due to the improved chemical, thermal and mechanical stability of catalyst materials printed on 3D carbon/ceramic supports. The enlarged high surface area controlled by the designed models enhanced the recycle and stability properties. (iv) By bridging the emerging printed electronics technology with a low-temperature chemical engraving method, nanostructured CuxO was in-situ constructed on well-designed ceramics skeleton. The obtained all-solid-state supercapacitors connected in series or parallel showed excellent mechanical properties and applicability as a new generation of mechanics required power sources.

Topic: 6 - Additive manufacturing of CMCs : 3D printing, laser sintering, etc.
Abstract no. 1906

# Laser assisted direct consolidation of CMC


Presenting author: M KUNZ Willy
Affiliation: Fraunhofer Institute for Ceramic Technologies and Systems IKTS
Winterbergstrasse 29,01277 Dresden (Saxony, Germany)

Co-authors: POLENZ Stefan (Fraunhofer Institute for Material and Beam Technology IWS), RößLER Marco (Fraunhofer Institute for Material and Beam Technology IWS), MICHAELIS Alexander (Fraunhofer Institute for Ceramic Technologies and Systems IKTS)



Additive manufacturing of CMCs is predicted to offer new possibilities regarding the decrease of fabrication time, the reduction of the amount of wasted materials and stress oriented fiber alignment. Comparable to the additive manufacturing of monolithic ceramics, several new approaches are upcoming, having different chances to potentially meet the requirements of industrial processes. One key-issue, missed by many processes, is the dispensability of time and energy consuming heat-treatments.

A new approach of laser assisted in-situ consolidation of CMCs is expected to overcome this drawback. Investigations on the interaction of laser radiation with potential matrix materials and different fibers are presented as well as first studies on the consolidation of composites. It has been found out that wavelength and matrix composition play a key-role in the feasibility of this process.

Topic: 6 - Additive manufacturing of CMCs : 3D printing, laser sintering, etc.
Abstract no. 2055

# Additive Manufacturing of Silicon Carbide parts


Presenting author: M ARBORE Romain
Affiliation: CTTC
7 rue soyouz,87068 Limoges (France)
Co-authors: BALLESTERO Anthony (CTTC), TOURNIE Hélène (CTTC), MAYAUDON Alexis (CTTC), BOYER Laurence (CTTC), DURAND Olivier (CTTC)



Silicon carbide is a non-oxide ceramic which has high mechanical properties, chemical inertia and good thermal resistance making it a perfect candidate for many applications (space, aeronautics, medical, filtration membranes, …). The main limit of silicon carbide is the shaping process and the machining step that is necessary to get the required dimensions. For reducing time and price of manufacturing and also to get easily more complex geometries, the use of additive manufacturing methods is a key point. At CTTC, Robocasting and Binder-Jetting technologies have been studied to explore the possibility to build near net shape parts.
Robocasting uses a plastic paste that is extruded through a moving nozzle, to build an object by a continuous shaping, layer by layer. First of all, a silicon carbide paste was formulated, in order to get a suitable feedstock for the Robocasting process. Various adjustments of the paste were necessary to optimize its rheological behaviour. Then, manufacturing parameters were studied and adapted to print flawless parts and finally thermal treatments allowed to obtain sintered parts with good quality.
Binder-jetting is a technique that consists to build a part layer by layer, by printing an organic binder through an inkjet head on a powder bed. With this printing method, no support is required, this function being assumed by the powder bed itself which allows to create complex structure. To succeed obtaining 3D parts, we both worked on the powder properties (flowability, grain size) and the printing parameters. The sintered silicon carbide pieces show a high level of porosity and nonetheless good mechanical properties.
This talk, focused on feedstock and powder formulations as well as additive manufacturing, will show the potential of these printing technologies for the manufacturing of silicon carbide pieces.

Topic: 6 - Additive manufacturing of CMCs : 3D printing, laser sintering, etc.
Abstract no. 2203

# Directed Energy Deposition of eutectic oxide ceramics: from powder to near net shape parts

Presenting author: Mme ODINOT Julie
Affiliation: DMAS, ONERA, Université Paris Saclay, F-92322, Châtillon, France
29 avenue de la Division Leclerc,92320 Châtillon (France)

Co-authors: CHORON Damien (IREPA LASER Illkirch France), JULIAN-JANKOWIAK Aurélie (DMAS ONERA Université Paris Saclay F-92322 Châtillon France), PETIT Johan (DMAS ONERA Université Paris Saclay F-92322 Châtillon France), BOISSELIER Didier (IREPA LASER Illkirch France), THOMAS Marc (DMAS ONERA Université Paris Saclay F-92322 Châtillon France)

Advanced ceramics are lightweight materials with an increasing interest for aerospace applications, such as airplane engines, exhaust components and satellite parts, owing to their chemical stability, erosion resistance and high temperature withstanding. However, such materials are subjected to manufacturing issues, especially when complex shapes are required. Therefore, the aim of this work is to investigate a new processing route using additive manufacturing for oxide-based ceramics.
In this study, DED-CLAD® process (Directed Energy Deposition – Construction with Laser Additive Direct process) is being developed for Al2O3-Y2O3-ZrO2 ternary eutectic. Powder flowability, laser absorption and thermal stress management are key points to adapt this process to ceramic materials, and then to achieve sound ceramic parts.
Flowable powders granules were designed by spray drying, and optical absorption of aforementioned composition has been increased by the addition of doping ions. Thanks to these achievements and to the implementation of complementary heating devices, ceramic samples have been successfully processed.
Now, material characteristics are under investigation as a function of manufacturing conditions. Different thermal cases, such as pre-heating, post-heating and layers remelting during construction led to differences between samples in terms of cracking, geometry and microstructures. The presence of cracks and porosity is being discussed with microstructure observations. Samples showed interesting nanometric-scale interconnected eutectic microstructures with growing size gradients depending on thermal history of the sample. Hardness, Young's modulus and fracture toughness are being evaluated thanks to instrumented nano and micro-indentation tests as well as flexure and compressive strength.
In parallel, optical absorption and thermal emissivity have been assessed. All these material characteristics are used as input parameters for process modelling using COMSOL Multiphysics. This model will help to better understand thermal stresses during manufacturing and to optimize process parameters as well as predicting the material's behaviour.

Topic: 6 - Additive manufacturing of CMCs : 3D printing, laser sintering, etc.
Abstract no. 1726

# The influence of sintering temperature on microstructure and properties of Al2O3 ceramic via 3D stereolithography printing technology


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| Presenting author: | Dr LI He |
| Affiliation: | Northwestern Polytechnical University<br>127 West Youyi Road,Beilin District, Xi'an Shaanxi,710072 Xi'an (Shaanxi, China) |
| Co-authors: | LIU Yongsheng (Northwestern Polytechnical University), LIU Yansong (Northwestern Polytechnical University), CHENG Laifei (Northwestern Polytechnical University), HU Kehui (Tsinghua University), LU Zhigang (Tsinghua University) |



Aluminum oxide (Al2O3) ceramic were fabricated by 3D stereolithography printing technology and subsequent debinding or sintering. In this work, the effect of sintering temperature on microstructure and properties were investigated. It was found that the flexure strength increased with the sintering temperature until reaching a maximum value of 148.8 MPa when sintered at 1500?. Furthermore, the shrinkage decreased with the decreasing sintering temperature until reaching a minimum value of 1.09% when sintered at 1200?. When sintered at 1280?, the flexure strength was 22.7 MPa and the shrinkage was 2.2% which meet the demands of ceramic core. It was evaluated that the higher sintering temperature lead to the greater shrinkage and larger crystallite size. Moreover, the energy spectrum analysis demonstrates that higher sinter temperature lead to higher O/Al atom ratio.

# Topic: 7

# Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)

Topic: 7 - Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)
Abstract no. 2064

# Oxidation in air plasma of ZrB2 and HfB2-SiC based composites from 1800 to 2200 K


Presenting author: Dr BALAT-PICHELIN Marianne
Affiliation: PROMES-CNRS laboratory
7 rue du four solaire,66120 Font-Romeu Odeillo (France)

Co-authors: RAPAUD Olivier (IRCER-CNRS laboratory), PIRIOU Cassandre (IRCER-CNRS), PELLEGRINI Charlène (PROMES-CNRS)



Fully-dense and microstructure-controlled zirconium and hafnium diborides were elaborated by Spark Plasma Sintering (SPS) with two different amounts of SiC – 20 and 30 vol.% – added to improve their oxidation resistance. A decrease of the sintering temperature with the amount of SiC was observed and directly connected to the removal of oxide impurities located at the surface of the diboride particles. In order to prevent any grain growth during the densification, sintering parameters were optimized by decreasing the imposed temperatures with the amount of SiC.

The oxidation in air plasma conditions from 1800 K up to 2200 K of these samples was studied using the MESOX facility implemented at the focus of the 6 kW Odeillo solar furnace. The mass variation of the samples during an oxidation duration of 300 s on a temperature plateau and in air plasma conditions at 1000 Pa total pressure was followed. From 1800 K up to nearly 2000 K, a positive mass gain was observed for all the samples due to the formation of a glassy phase. The mass loss is sensitive after 2000 K due to the partial vaporization of silica, SiO and CO and this differently according to the amount of SiC. Then since 2100 K, the mass loss is significant and tends to increase more slowly up to 2200 K.

A comparison with some experiments conducted in standard air will be presented.

SEM images, XRD characterizations and thermo-radiative properties were performed to understand the oxidation phenomena and to know the emissivity of these materials at high temperature. Some experimental results will be presented.

Topic: 7 - Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)
Abstract no. 2071

# The effect of PAN or pitch-based C fibers on the microstructure and properties of continuous Cf- ZrB2/SiC UHTCMCs


Presenting author: Dr SCITI Diletta
Affiliation: CNR-ISTEC
Via Granarolo 64,48018 Faenza (Japan)

Co-authors: ZOLI Luca (ISTEC-CNR), VINCI Antonio (CNR-ISTEC), SILVESTRONI Laura (CNR-ISTEC), GALIZIA Pietro (ISTEC-CNR), REIMER Thomas (Deutsches Zentrum für Luft- und Raumfahrt (DLR), RIVERA Sergio (Nanoker Research)



UHTCMCs combine good fracture toughness and thermal shock resistance of CMCs with oxidation and ablation resistance of UHTCs. Recently, we have shown that UHTCMCs can be produced by infiltration of carbon fiber preforms with aqueous suspensions of the ceramic powders followed by densification by Spark Plasma Sintering (SPS) (www.c3harme.eu).
In this talk we compare the microstructure and high temperature properties of two different ZrB2/SiC/Cf composites reinforced with continuous PyC coated PAN-derived fibres or uncoated pitch fibres. We up-scaled our process producing discs with diameter of 150-170 mm to obtain a high number of bars and evaluate different properties, including: flexural, tensile and compressive strength, fracture toughness and thermal shock resistance.
Ultra-high modulus pitch-based carbon fibres showed a lower degree of reaction with matrix grains during sintering compared to intermediate modulus PyC/PAN-derived fibres. The reason lies in the different microstructure of the carbon, highly crystalline with a graphitic structure in the former and amorphous with a turbostratic structure in the latter. The presence of a coating for PAN-derived fibres was found to be essential to limit the reaction at the fibre/matrix interface during SPS, However, coated bundles were more difficult to infiltrate, resulting in a less homogeneous microstructure.
As far as the mechanical properties are concerned, specimens reinforced with coated PAN-derived fibres provided higher strengths and damage tolerance than uncoated pitch-fibers, due to the higher degree of fibre pull-out and porosity. On the other hand, the weaker fibre/matrix interface resulted in lower interlaminar shear, off-axis, and compressive strength.

Topic: 7 - Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)
Abstract no. 1853

# Damage-tolerant ternary layered borides MAB phases: Theoretical and experimental insights


Presenting author: Prof BAI Yuelei
Affiliation: Harbin Institute of Technology
2# Yikuang Street,150080 Harbin (China)

Co-authors: QI Xinxin DUFF A.I. LI Ning SUN Dongdong HE Xiaodong ZHENG Yongting WANG Rongguo LEE W.E



Over the past two decades, a class of ternary transition metal carbides or nitrides known as MnAXn+1 phases (MAX phases for short where M is an early transition metal, A is a group IIIA or IVA element, X is C and/or N, and n = 1-3), formed by inserting A-group atoms into the corresponding binary carbides or nitrides, have attracted growing attention due to their unique combination of metallic and ceramic properties [1]. Analogous to the MAX phases, inserting Al layer/s into the binary borides indeed results in the formation of ternary layered borides called “MAB phases” [2], with recent experimental work on Fe2AlB2 [3] and MoAlB [4] (both MAB phases) showing high fracture toughness, damage tolerance and oxidation resistance.

Theoretically, the electronic structure, anisotropic compression, elastic properties, damage tolerance and fracture behavior were been well addressed from the MAX phases to the MAB phases. By use of the previously proposed theoretical model of “bond stiffness” [5], of more importance, it is revealed that the weak Al-Al or M-Al bonds in origin contribute to the experimentally observed damage tolerance and fracture toughness in the promising MAB phases [6] as UHTCs for ultra-high temperature. Furthermore, the different damage and fracture behavior of Cr-containing MAB phases was predicted, originating from the different bonding strength among nanolaminates in their different crystal structures [7]. Experimentally, the successful fabrication of polycrystalline Fe2AlB2 bulk for the first greatly reduced the fabrication time of MAB phases [3]. No dominant indentation cracks are observed, indicating that Fe2AlB2 may be quite damage tolerant. Interestingly, a noncatastrophic failure is present in the SENB test, with a high work of fracture (Figure 1). The brittle-ductile transition temperature (BDTT) is higher under flexure (>1000 oC) than compression (800-900 oC), which is attributed to the higher shear stress under compression [8].

Topic: 7 - Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)
Abstract no. 2091

# Retained strength of UHTCMCs treated above 2273 K in oxidizing environment


Presenting author: Dr MONTEVERDE Frederic
Affiliation: CNR-ISTEC
Via Granarolo,64,48018 Faenza (Italy)

Co-authors: SCITI Diletta (National Research Council of Italy Institute of Science and Technology for Ceramics Via Granarolo Faenza (RA), 64-48018 Italy), SILVESTRONI Laura (National Research Council of Italy Institute of Science and Technology for Ceramics Via Granarolo Faenza (RA), 64-48018 Italy.), ZOLI Luca (National Research Council of Italy Institute of Science and Technology for Ceramics Via Granarolo Faenza (RA), 64-48018 Italy.), SARAGA Federico (National Research Council of Italy Institute of Science and Technology for Ceramics Via Granarolo Faenza (RA), 64-48018 Italy.), VINCI Antonio (National Research Council of Italy Institute of Science and Technology for Ceramics Via Granarolo Faenza (RA), 64-48018 Italy.), GALIZIA Pietro (National Research Council of Italy Institute of Science and Technology for Ceramics Via Granarolo Faenza (RA), 64-48018 Italy.), REIMER Thomas (Institute of Structures and Design German Aerospace Center Stuttgart Germany



A new class of non-oxide ultra-high temperature ceramic-matrix composites (UHTCMCs), currently under study within the Horizon 2020 European C3HARME research project, is attracting a extensive interest. One of the key-features such class of materials should be able to perform is self-repair damage without any external intervention, widely known as self-healing. The self-healing capability in UHTCMCs can be implemented by tailoring additions of ultrafine substances that, under the contemporary effect of temperature and oxygen content typical of the launch or re-entry phases, trigger the formation of protective layers capable of fixing original defects or new ones generated by the severe operating service. This technological achievement vastly improves the reliability of the structural ceramic components and make materials reusable, significantly reducing costs of space missions.
To assess the self-healing capability, room temperature strength was measured using samples variously treated (i.e. as produced and after heat conditioning above 2273 K) to determine corresponding recovery strength rates. The effects of the service ambient on various ceramic matrices constituting different UHTCMCs were separated and evaluated.

Topic: 7 - Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)
Abstract no. 1827

# Influence of composition and grain size on the damage evolution in MAX phases investigated by acoustic emission

Presenting author: Dr ANTOU Guy
Affiliation: Univ of Limoges - IRCER (UMR CNRS 7315)
12 rue Atlantis,Centre Européen de la Céramique,87068 Limoges (France)

Co-authors: ANTOU Guy (Univ of Limoges - IRCER (UMR CNRS 7315), CHOTARD Thierry (Univ of Limoges - IRCER (UMR CNRS 7315), LIS Jersy (AGH UST 30-059 Cracow Poland)

In this study, the influence of grain size and phase composition on damage mechanisms of $Ti_3SiC_2$-based materials are investigated. Commercially available and self-propagating high-temperature synthesized powders were sintered via spark plasma sintering and hot pressing methods. Materials with different amounts of $Ti_3SiC_2$ (from 52 to 72 wt. %) and various mean grain sizes (from 8 to 20 µm) were characterized by performing bending tests coupled with acoustic emission measurements. It permits to distinguish the involved damage mechanisms and their chronology in MAX phase-based materials. With the increase of the applied stress, damage begins with the onset of delaminations within MAX phase grains, then friction processes occur within previously formed microcracks, and finally (before rupture) new microcracks are generated due to the elasticity mismatch between phases. Increasing $Ti_3SiC_2$ content and grain size emphasizes the two first damage stages, and finally leads to a more pronounced nonlinear behavior.

Topic: 7 - Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)
Abstract no. 2062

# PROTECTIONS AGAINST OXIDATION, BY CVD,SPS AND RMI OF HAFNIUM CARBIDE AND SILICON CARBIDE, ON CARBON/CARBON COMPOSITES.

Presenting author: Dr ALLEMAND ALEXANDRE
Affiliation: CEA
3 Allée de la Boetie,33600 PESSAC (France)
Co-authors: LE PETITCORPS YANN (UNIVERSITE BORDEAUX-LCTS), JACQUES SYLVAIN (CNRS-LCTS), VERDON CLAIRE (CNRS-LCTS), SZWEDEK OLIVIER (CEA-LCTS), GUILLERMIN EDOUARD (CNRS-LCTS)

The hafnium carbide compound is an ultra high refractory ceramic; as a result it could be of interest for the protection of carbon/carbon composites against oxidation at high temperatures. However HfC a present a non stoechiometric composition with carbon vacancies. As a consequence, the oxidation resistance is poor at low temperatures (500-1000°C). In order to overcome this main drawback the HfC can be associated with silicon carbide (SiC).
Three coating or infiltration routes have been studied; the first one is the Chemical Vapour Deposition which enables to obtain very thin coatings, the second one is the Spark Plasma Sintering technique which permits to get new microstructures and the third one is the Reactive Melting Infiltration of HfSi2.
On first hand, the CVD enables to get an alternated multilayer microstructure made of a first layer of SiC on top of which the first layer of HfC is deposited and so on to a ten alternated layer deposit [1].
In a second hand, SPS has permitted to sinter, on carbon substrate, ultra high refractory ceramic powders with a significant amount of SiC. The fluidized bed CVD is used to deposit a layer of SiC on top of HfC grains. The powder obtained has a core shell structure [2].
In a third way, R M I enables, at the fusion temperature of HfSi2 (ie 1800°C), to get with reaction of the liquid and C inside the sample an alternated layer of SiC and HfC inside a dense 3D C/C. By repeating this, an infiltrated multilayer can be obtained [3].
To achieve this study those C/C coated have been tested up to 2000°C under air in an arc image furnace at a very high heating rate. The coatings obtained by SPS had been used to understand the oxidation mechanism involved during oxidation [4].

Topic: 7 - Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)
Abstract no. 2095

# Fabrication and characterization of UHTCMCs based on the ZrB2/SiC/Y2O3 system


Presenting author: Dr VINCI Antonio
Affiliation: ISTEC - CNR
Via Granarolo, 64,48018 Faenza (Ra, Italy)

Co-authors: ZOLI Luca (ISTEC - CNR), GALIZIA Pietro (ISTEC - CNR), SILVESTRONI Laura (ISTEC - CNR), SCITI Diletta (ISTEC - CNR), SAVINO Raffaele (University of Naples Federico II), MUNGIGUERRA Stefano (University of Naples Federico II)



The materials currently used in extreme environments, based on C/C or C/SiC composites, have good mechanical properties but are not suitable for temperatures above 2000°C. Ultra-high temperature ceramics (UHTCs) have been gaining increasing interest as potential replacements owing to their high melting points (> 3000°C) and high temperature strength but their low fracture toughness and thermal shock resistance remains a major concern.
To overcome these limits, we are currently designing and manufacturing a new class of composites based on CMCs with a UHTC matrix in the framework of the European project C3HARME.
In this work we investigate the effect of Y2O3 addition on the oxidation resistance and mechanical properties of carbon fibre reinforced ZrB2/SiC composites. SiC is known to provide oxidation resistance up to 1650°C, but beyond this temperature, the protective silica layer starts evaporating leaving behind a porous ZrO2 scale. To further improve the oxidation resistance, Y2O3 was added to promote the formation of a compact ZrO2 scale.
Specimens were produced by slurry infiltration and hot pressing. From SEM analysis on the sintered specimen, fibres were found homogeneously dispersed in the ceramic matrix which was fully dense. Y2O3 reacted both in the ceramic matrix and at the fibre/matrix interface, forming lamellar YCBO phases.
The sample was tested in an arc-jet facility at 2400 K and survived the erosion test, registering an erosion rate of only ~10^-4 mm/s. Further analysis on the surface and microstructure of the oxidized sample revealed the formation of an outer compact layer of ZrO2, an intermediate layer of ZrO2/SiO2 and the unreacted composite.
Bending tests were carried out on the composite to evaluate the flexural strength and fracture toughness. The addition of Y2O3 resulted in a considerable improvement of the mechanical resistance at room and high temperature compared to an un-doped ZrB2/SiC composite used as reference.

Topic: 7 - Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)
Abstract no. 2273

# Observing the formation of high entropy metal diborides and studying the influence of single boride in the solid solution using arc-melting as consolidation technique.


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| Presenting author: | M FAILLA Simone |
| Affiliation: | National Research Council of Italy - Institute of Science and Technology for Ceramics (CNR-ISTEC)<br>Via Granarolo 64,48018 Faenza (Italy, Italy) |
| Co-authors: | SCITI Diletta (National Research Council of Italy - Institute of Science and Technology for Ceramics (CNR-ISTEC), GRASSO Salvatore (Key Laboratory of Advanced Technologies of Materials Ministry of Education School of Materials Science and Engineering Southwest Jiaotong University), FU Shuai (Laboratory of Advanced Technologies of Materials Ministry of Education School of Materials Science and Engineering Southwest Jiaotong University) |



In the last few years, processing of high entropy metal diborides has been studied by many researchers, mainly through spark plasma sintering or hot pressing at temperatures over 2000°C. The biggest challenge is the formation of a homogeneous solid solution, and prediction of the multiple combinations that are more likely to result in a single phase. Arc-melting is a ultra-fast consolidation method, that allows to reach temperatures well above the melting point (>3000 °C) of UHTCs.
In this work, arc-melting was used to directly synthesize/sinter high entropy metal diborides mixing $HfB_2$, $ZrB_2$, $TiB_2$ $TaB_2$ and $CrB_2$. In order to understand the influence of each individual boride, 5 equimolar combinations of 4 out of 5 borides were studied; e.g Hf-Zr-Ti-Ta, Hf-Zr-Ti-Cr, Hf-Zr-Ta-Cr, Hf-Ti-Ta-Cr, Zr-Ti-Ta-Cr. Arc-melting allowed a fast discrimination of favorable and unfavorable combinations. For instance, Cr was hardly found in solid solution, as it preferably segregated at the grain boundaries. Vickers hardness and oxidation tests at 1600°C in air were carried out and results were compared with conventional monophasic borides.

Topic: 7 - Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)
Abstract no. 1863

# Continuous carbon fibre reinforced ZrC composites with SiC interphase and rare earth oxide dopants


Presenting author: Dr TAMMANA Sree Rama Chandra Murthy
Affiliation: School of Metallurgy and Materials, University of Birmingham
C/o. Prof. Jon Binner, 1B20,School of Metallurgy and Materials,B15 2SE
Birmingham (west Midlands, United Kingdom)
Co-authors: VENKATACHALAM Vinothini (University of Birmingham), BINNER Jon (University of Birmingham)



Ultra-high temperature ceramic materials are candidates for a variety of high-temperature structural applications, including rocket engines, hypersonic vehicles, plasma arc electrodes, advanced nuclear fission / fusion reactor components, cutting tools, furnace elements and high-temperature shielding. Zirconium carbide (ZrC) is considered to have good potential candidate due to its high melting point and good chemical inertness, but the monolithic form suffers from poor fracture toughness, oxidation and thermal shock resistance. Hence, reinforcing it using continuous carbon fibres to create an ultra-high temperature ceramic matrix composite (UHTC) has been explored. To enhance the fracture toughness and thermal shock resistance, tailored interphases between the fibres and matrix need to be developed, whilst oxidation and ablation resistance can be improved by doping with suitable additives. However, due to the very high sintering temperature of ZrC, densification is challenging without the use of very high temperatures combined with significant pressure, e.g. by conventional hot pressing or spark plasma sintering. Use of these techniques, however, risks damaging the fibres. In the present work, therefore, radio frequency assisted chemical vapour infiltration (RF-CVI) has been used to make 2.5D Cf reinforced ZrC matrix composites. A suitable interphase of SiC combined with rare earth oxide (CeO2 & Y2O3) dopants were also incorporated to enhance the fracture toughness, thermal shock resistance and thermal properties. Results on the processing parameters, microstructural characterisation, oxidation and ablation properties of the composites will be presented.

Topic: 7 - Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)
Abstract no. 1968

# Fabrication and performance of ultra-high temperature ceramic matrix composites through RF enhanced chemical vapour infiltration


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| Presenting author: | Dr VENKATACHALAM Vinothini |
| Affiliation: | Postdoctoral Researcher<br>School of metallurgy and materials,University of Birmingham,B15 2TT Birmingham (West midlands, United Kingdom) |
| Co-authors: | BINNER Jon (Professor Deputy Head of College College of Engineering and Physical Sciences) |



Ultra-high temperature ceramics (UHTCs) are typically transition metal borides and carbides, though the boride-based UHTCs are often preferred because their oxidation resistance is greater. They are typical ceramics, however, and so in their monolithic form they are brittle materials that fail catastrophically when their strength or thermal shock resistance is exceeded. Reinforcing them with carbon fibers can increase their toughness and hence thermal shock behaviour significantly, whilst the presence of the UHTC phase protects the carbon fibres from oxidation at the application temperatures. Thus ultra-high temperature ceramic matrix composites, UHTCMCs, have excellent potential for use in aerospace applications such as rocket nozzle throats and thermal protection systems.

The composites being developed at the University of Birmingham within the European Union-funded, Horizon 2020 research programme known as C3Harme, are based on 3 discrete phases, viz. carbon fibres impregnated with zirconium diboride and carbon matrix. The latter is deposited within the fibre / powder preform using chemical vapour infiltration (CVI). This approach, which operates at temperatures as low as about 1000oC, has the advantage of minimising damage to the fibres and is capable of producing near net shape components along with uniform, cohesive and conformal matrices. However, the downside of conventional CVI is the very long processing time required as it relies on heating from outside and hence very slow heating rates are needed to avoid the pore entrances at the surface of the component becoming blocked. Switching to radio frequency (RF) assisted heating means that the samples are heated from the inside out, avoiding the gas channels becoming blocked. Process times can be reduced by more than an order of magnitude as a consequence. This presentation will report on the latest results from the research including the control of the fibre to matrix interphase, which plays a key role in improving the thermomechanical properties.

Topic: 7 - Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)
Abstract no. 2248

# High-Temperature Mechanical Characterization of UHTCMCs


Presenting author: M REIMER Thomas
Affiliation: Deutsches Zentrum für Luft- und Raumfahrt (DLR)
Pfaffenwaldring 38-40,70569 Stuttgart (Germany)

Co-authors: SCITI Diletta (CNR-ISTEC), KOCH Dietmar (Deutsches Zentrum für Luft- und Raumfahrt (DLR), KüTEMEYER Marius (Deutsches Zentrum für Luft- und Raumfahrt (DLR), ZOLI Luca (CNR-ISTEC), VINCI Antonio (CNR-ISTEC), SILVESTRONI Laura (CNR-ISTEC), GALIZIA Pietro (CNR-ISTEC), MONTEVERDE Frederic (CNR-ISTEC), RIVERA Sergio (Nanoker)



UHTCMCs promise to combine good fracture toughness and thermal shock resistance of CMCs with oxidation and ablation resistance of UHTCs. Recently, the C3HARME project funded by EU (www.c3harme.eu) has taken to advance the processing technology of UHTCMC materials to arrive at components which can be used in critical space applications like thermal protection systems of re-entry vehicles or nozzle throats of rocket motors.

In the material development a number of different compositions of material constituents have been investigated. In order to evaluate the quality of the materials, mechanical characterization of samples was an important part of the work. A wide range of characteristic properties were determined, e.g. strength, toughness, modulus and more. Mechanical tests were done also at high temperature.

In terms of high temperature mechanical characterization, bending as well as tensile tests were carried out. The tests were conducted in the high-temperature test facility INDUTHERM at DLR. The facility is a mechanical test machine upgraded for tests at very high temperature. The heating is done via induction heating of graphite susceptors by a HF generator and coil and indirect heating of the samples via radiation. The facility is equipped with a water-cooled test chamber to have low pressure or an inert gas environment. Test temperatures are in principal only limited by the materials used for the supports and clamps. Typical test temperatures are in the range of 1600 - 1800°C for bending and tensile tests. An effort will be made in the near future to increase the test temperature to 2000°C for such tests.

In this talk results will be presented on high-temperature bending and tensile tests with special consideration of the testing infrastructure. Also briefly presented will be a setup that was developed to oxidise samples in a controlled way at ultra-high temperatures above 2000°C without mechanical load.

Topic: 7 - Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)
Abstract no. 1966

# Experimental investigation on sintered UHT-CMC composites for Combustion Harsh Environments and Space


Presenting author: M MUNGIGUERRA Stefano
Affiliation: University of Naples "Federico II"
Piazzale Tecchio 80,80125 Naples (Italy)

Co-authors: "DI MARTINO Giuseppe Daniele ( University of Naples "Federico II"), CECERE Anselmo ( University of Naples "Federico II"), SAVINO Raffaele ( University of Naples "Federico II"), ZOLI Luca ( CNR-ISTEC), VINCI Antonio ( CNR-ISTEC), SILVESTRONI Laura ( CNR-ISTEC), SCITI Diletta ( CNR-ISTEC)



Ultra-High-Temperature Ceramic (UHTC) composites, based on early transition metals carbides and diborides, as major phases, and silicon carbide, as minor phase, thanks to their high melting temperatures, strength and oxidation resistance at temperatures over 2000°C, are considered appealing candidates for aerospace applications involving extreme aero-thermo-chemical environments, such as reusable Thermal Protection Systems (TPS) for hypersonic vehicles and erosion-resistant rocket nozzles. Since monolithic materials demonstrated low fracture toughness, low thermal shock resistance and lack of damage tolerance, fiber-reinforced Ultra-High-Temperature Ceramic Matrix Composites (UHT-CMC) are currently regarded as the most promising technology for overcoming state-of-art materials limitations. Hence, the Horizon 2020 European C3HARME research project is focused on a new class of UHT-CMCs for near zero-erosion rocket nozzles and near zero-ablation TPS. Extensive experimental characterization campaigns are ongoing, based on an incremental approach, envisaging tests on prototypes of increasing complexity.

Sintered $ZrB_2$-based ceramics reinforced with carbon fibers are characterized in two main facilities: a supersonic arc-jet wind tunnel, where atmospheric re-entry conditions are reproduced at maximum flow total enthalpies higher than 20 MJ/kg, supersonic Mach number and temperatures over 2000°C in a gas atmosphere with high concentration of atomic oxygen; and a lab-scale hybrid rocket engine, where temperatures over 3000 K, chamber pressure on the order of 10 bar and a considerably oxidizing environment allow to reproduce a representative operating environment. Experimental results, including non-intrusive infrared temperature diagnostics and ablation/erosion measurements, are analyzed in order to compare different UHTCMC formulations and investigate the effect of factors such as carbon fibers architecture and porosity level on material performance.

Post-test inspections are carried out to analyse the micro-scale surface modifications occurred after the exposition to the aero-thermo-chemically aggressive flows. Moreover, one-

dimensional models based on chemical equilibrium and computational fluid dynamic models are defined and employed to provide useful data for interpretation of the experimental results.

Topic: 7 - Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)
Abstract no. 1980

# Carbosilicothermic synthesis of MAX phases and high-entropy MX carbides


Presenting author: Dr ISTOMIN Pavel
Affiliation: Institute of Chemistry, Komi Science Centre of the Ural Branch of the Russian Academy of Sciences
Pervomaiskaya, 48,167982 Syktyvkar (Russia)

Co-authors: ISTOMINA Elena (Institute of Chemistry Komi Science Centre of the Ural Branch of the Russian Academy of Sciences), NADUTKIN Aleksandr (Institute of Chemistry Komi Science Centre of the Ural Branch of the Russian Academy of Sciences), GRASS Vladislav (Institute of Chemistry Komi Science Centre of the Ural Branch of the Russian Academy of Sciences), SHELUD'KO Svetlana (Institute of Chemistry Komi Science Centre of the Ural Branch of the Russian Academy of Sciences), OPLESNINA Elizaveta (Institute of Chemistry Komi Science Centre of the Ural Branch of the Russian Academy of Sciences), DMITRIEVA Inga (Syktyvkar State University)



MAX phases of the groups 4 and 5 transition metals are of great interest for high-temperature engineering applications because of their unique combination of properties, such as thermal shock resistance, high fracture toughness, damage tolerance, chemical resistance, etc. A novel approach to the synthesis of these compounds via the vacuum carbosilicothermic reduction (VCSTR) of transition metal oxides is presented herein. The essence of the proposed approach is that it combines in one pot carbothermic and silicothermic reduction processes through the use of SiC as a reductant. This makes it possible to synthesize silicon-containing MAX phases. In particular, the VCSTR method has been successfully applied for the synthesis of Ti4SiC3 MAX phase in bulk form, which could not be obtained by conventional methods. It was also found that the VCSTR method can be used for obtaining MAX phase solid solutions in the Ti-(Me)-Si-C systems, where M = Zr, V, and Nb.

High-entropy MX carbides of the groups 4 and 5 transition metals are yet another promising material for ultra-high temperature applications. It was demonstrated that the VCSTR method is well suited for the synthesis of these compounds. In particular, (Ti0.2Zr0.2Hf0.2Nb0.2Ta0.2)C has been obtained by this method.



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Topic: 7 - Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)
Abstract no. 1914

# Development and characterization of ultra-high temperature ceramics matrix composites


Presenting author: Dr JUSTIN Jean-François
Affiliation: ONERA
29 avenue de la division Leclerc,92322 Châtillon (France)

Co-authors: DEBARRE Antoine (DMAS-ONERA), JULIAN-JANKOWIAK Aurélie (DMAS-ONERA)



Ultra-High Temperature Ceramics (UHTC) are a class of materials of growing interest for high temperatures applications (>2000°C) in corrosive and/or oxidising atmospheres. $ZrB_2$-SiC and $HfB_2$-SiC-based compositions are ones of the most studied in the literature as they exhibit very high melting points (>3200°C) and good thermal shock resistance. Oxidation behaviour of this class of materials have been extensively studied demonstrating the formation of a multi-oxide scale composed of a $MeO_2$ skeleton and a glassy borosilicate layer at high temperatures. Since more than a decade, Onera has carried out several activities on UHTC materials from manufacturing to thermomechanical assessments of monolithic components in realistic environment and oxidation resistance understanding. Last developments are centred on the manufacturing of ultrahigh temperature ceramics matrix composites (UHTCMC).

Slurry infiltration and sintering was selected to manufacture UHTCMC reinforced with carbon fibres fabrics. First works were focused on the study of the stability of aqueous slurries containing ZrC or HfC powders. The main objective was to reach the higher ceramic contents with the lower viscosity possible. Thus, different slurries, prepared with four kinds of dispersive agents and/or pH, were assessed through viscosity measurements. Therefore, slurries with 45-55 vol. % of powder were optimised for the infiltration process. The second part of the work concerned the infiltration step. The selected carbon fabrics were highly permeable to make the infiltration process easier and the volume of the mold was fixed. After infiltration, samples were hot pressed at 1500°C to sinter the matrix without degrading the fibres. Microstructural characterisations show a good infiltration of the matrix in the tows without any reaction with the fibres. The third part focused on the C interphase coating. Polymer infiltration and pyrolysis process was first considered but the coating was inhomogeneous and mechanical properties were low. Chemical Vapor Deposition was finally investigated to improve the UHTCMC.

Topic: 7 - Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)
Abstract no. 2297

# Accelerated development of MAX phase-coated accident-tolerant fuel cladding materials (invited)

Presenting author: Prof LAMBRINOU Konstantina
Affiliation: SCK-CEN
Boeretang 200,2400 Mol (Belgium)

Co-authors:

This lecture presents the challenges involved in the accelerated development of accident-tolerant fuel (ATF) cladding materials coated with MAX phases. ATF development is one of the greatest current pursuits in the nuclear sector: it aims at improving nuclear energy safety in the post-Fukushima era by developing fuel cladding concepts that can overcome the inherent technical shortcomings of standard zircaloy/UO2 nuclear fuels. The accelerated development of innovative ATF clads must ensure the constant communication between application-driven material design, material production, and material performance assessment in application-relevant conditions. Successfully implementing this approach relies on the development of high-throughput material screening tools capable of assessing material performance prior to in-pile testing; one such tool involves the use of ion/proton irradiation to assess material radiation tolerance.

The MAX phases are nanolayered ternary carbides and nitrides given by the Mn+1AXn general formula, where M is an early transition metal, A is an A-group element, X is C or N, and n = 1, 2 or 3. This lecture addresses the stepwise development of MAX phase coatings on commercial zircaloy-4 (Zry-4) substrates, thus improving the compatibility of the latter with the coolant (steam) under transient/accidental operating conditions. The Cr2AlC MAX phase coatings were deposited on Zry-4 substrates by magnetron sputtering. The coated systems were designed to include bi-layer diffusion barriers between Zry-4 substrate and Cr2AlC; the diffusion barriers were defined by thermodynamic modelling and prevented coating/substrate reactivity during high-temperature accidents. The compatibility of these coatings with the coolant (i.e., water, steam) was assessed by dedicated aqueous corrosion and steam oxidation tests. Moreover, the response of the coatings to irradiation was studied by means of in-situ ion irradiation in the TEM: this monitored dynamic evolution of damage and showed the importance of the coating microstructure (grain size, texture) on the radiation tolerance of the coated system.

Topic: 7 - Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)
Abstract no. 2178

# On the anisotropic thermal performance of MAX phase matrix composites with carbon reinforcements. The case of Nb2AlC/CF


Presenting author: Dr AZINA Clio
Affiliation: Linköping University
Linköping University, Campus Valla,58381 Linköping (Sweden)

Co-authors: ROSéN Johanna (Linköping University - Department of Physics Chemistry and Biology), ROGER Jérôme (Laboratoire des Composites ThermoStructuraux), BATTAGLIA Jean-Luc (Institut de mécanique et d'ingénierie - Université de Bordeaux), EKLUND Per (Linköping University - Department of Physics Chemistry and Biology)



In the attempt to produce material systems able to withstand high temperatures, mechanical solicitation and function in harsh environments, MAX phases have gained attention through the years. Their unique combination of metallic and ceramic properties makes them suitable candidates for structural materials, among others. By combining the highly anisotropic structure of these compounds with anisotropic reinforcements, one can expect that the resulting composite material will exhibit highly anisotropic properties, which can be tailored to suit the destined application. In this case, interest is brought to the thermal performance of MAX phases and their composites. To this end, Nb2AlC MAX phase powders were synthesised through pressureless sintering, in order to produce the matrix material. The powders, constituted of ~80 wt.% of pure MAX phase and ~20 wt.% of a mixture of secondary phases (Nb4AlC3 and NbC), were grinded to an average of 44 ?m to match the dimensions of the reinforcements, for homogeneous mixing. Carbon fibres are used as reinforcements for their high thermal conductivity along the fibre axis. The composite materials were sintered using Spark Plasma Sintering, at high temperature and under load in order to promote the alignment of the reinforcements perpendicular to the pressure axis and produce highly oriented composite materials. Microstructural and chemical analyses were carried out at the interfacial regions between matrix and reinforcements, while the flash method was used to determine the thermal diffusivity of the composite materials at different temperatures.

Topic: 7 - Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)
Abstract no. 1874

# The mechanism of the interaction of iridium with refractory borides and carbides


Presenting author: Prof BAKLANOVA Nataliya I.
Affiliation: Institute of Solid State Chemistry and Mechanochemistry SB RAS
Kutateladze st.18,630128 Novosibirsk (Russia)

Co-authors:



The development of structural materials for use in oxidizing environments at temperatures above 2000°C is of great interest to the aerospace sector. Materials for these environments are currently largely limited to ultra-high temperature ceramics based on borides and carbides of transition metals of IV-V group. Borides and carbides have melting temperatures close to or above 3000°C and retain strength and thermal shock resistance at moderate temperatures. Intermetallics may be of interest as secondary phase to improve the corrosion resistance of borides and carbides. As a rule, the most suitable intermetallic compositions are those combining iridium with hafnium and tantalum. Iridium has low recession rate in oxidative atmospheres at temperatures as high as 2000?C and hafnium and tantalum oxide have high melting points and low vapor pressures at high temperatures.

The aim of this study was to investigate the mechanism of solid-state interaction of iridium with tantalum and hafnium carbides, as well as with corresponding borides. The solid-state interaction of Ir with refractory carbides (borides) was performed by two techniques: (i) hot pressing of corresponding powder mixtures and (ii) heat-treatment of contact couples consisted of iridium foil and sintered ceramic disks at different temperatures. It was shown that the interaction of Ir and carbides leads to the formation of the only intermetallic product Ta(Hf)Ir3. In addition, a free carbon phase of complex morphology is released. The mechanism of boride-iridium solid-state interaction is different one. The new ternary Hf(Ta)-Ir-B phases were formed. The capability of the iridium-ceramics systems to withstand the extreme environmental conditions was evaluated.



This work was supported by the Russian Science Foundation, Grant #18-1900075.

Topic: 7 - Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)
Abstract no. 2167

# Synthesis of high purity ultra-fine HfC, HfB2 powder – effects of carbon source and drying methods


Presenting author: Dr KIM JUNSEOP

Affiliation: Korea Institute of Materials Science
797, Changwondaero, Seongsangu,51508 Changwon (Gyeongsangnam-do, South Korea)

Co-authors: HEE JUNG Lee (Powder & Ceramics Division/ Korea Institute of Materials Science), SEA-HOON Lee (Powder & Ceramics Division/ Korea Institute of Materials Science)



Ultra-fine High purity hafnium carbide (HfC) and Hafnium diboride(HfB2) powder were synthesized from organic-inorganic hybrid slurry based on carbothermal reduction(CTR) and Borocarbothermal reduction (BCTR) process using the modified spark plasma sintering (SPS) apparatus. Hafnium dioxide(HfO2), Phenolic resin, Sucrose, and Boric acid used as a source of Hafnium, carbon and boron source, respectively. The effects of dispersion treatment of precursor slurry, kind of carbon source, and drying method on the purity and size of the final powder have been investigated. Ultra-fine (about 100nm) and high purity (>99.9% except for Zr) HfC and HfB2 powders were obtained after the CTR and BCTR at ~1600? for 1hr. The amount of residual carbon and oxygen in the final powder could be reduced by the de-agglomeration of HfO2 powder. The amount of residual carbon and oxygen in the final powder could be minimized by improving mixing uniformity during the drying process through rotation drying and freeze drying. By using sucrose - distilled water system instead of phenol resin – ethanol system as a carbon source, a smaller and more uniform powder could be synthesized with less toxic and environmentally friendly processes. XRD, SEM, and TEM confirm the fine particle size and uniform distribution of HfC and HfB2 powder. HfC and HfB2 ceramics with a high relative density (>98%) were obtained after the sintering of the synthesized powder with ~ 2000? and ~80MPa pressure. The average grain size of the sintered specimen was about 7µm. This process is a promising process for commercialization of UHTC powder synthesis.

Topic: 7 - Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)
Abstract no. 1716

# Morphology and Anti-Ablation Properties of the Precursor Infiltration & Pyrolysis Cf/C-SiC Composites under Oxy-Acetylene Test


Presenting author: Dr ZHU YANG
Affiliation: Xi'an Aerospace Composites Research Institute
Tianhong street, No. 1,Baqiao district,710025 Xi'an (SHAANXI, China)

Co-authors: YAN Liansheng (Xi'an Aerospace Composites Research Institute), CUI Hong (Xi'an Aerospace Composites Research Institute), WEI Huali (Xi'an Aerospace Composites Research Institute)



In this work, the needled carbon fiber preforms were used to make seven groups of carbon/carbon (C/C) composite billets by controlling the process time of chemical vapor infiltration (CVI) to form different matrix carbon contents. These C/C composites were then modified by polycarbosilane (PCS) through precursor infiltration and pyrolysis (PIP) repeatedly until the mass clamming between two cycles is less than 3% of the initial weight (above 1.9g/cm3) to make Cf/C-SiC composites. These specimens were tested under the oxy-acetylene firing environment (according to GJB 323A-96, heat flux around 4200kW/m2) for 200s, 300s and 400s, respectively, finding that higher content of PIP ceramic matrix had enhanced the anti-ablation properties of the composite. Besides that, specimens bearing longer duration tests had a trend of lower average ablation rates. The lowest linear ablation rate is 0.008mm/s and the mass ablation rate is 0.0019g/s for those high SiC content specimens tested for 400s. The SEM images of the tested samples showed the mechanisms and the step by step process of the ablative resistance progress.

Topic: 7 - Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)
Abstract no. 2441

# Fabrication of C/C-ZrC composites using LMI route and their evaluation under solid rocket motor exhaust plume conditions


Presenting author: Dr KUMAR Suresh
Affiliation: DMSRDE
GT Road,Kanpur,208013 Kanpur (Uttar Pradesh, India)

Co-authors: KHAN Bablu KUMAR Satyendra PRASAD N Eswar



Carbon fiber reinforced zirconium carbide (C/C-ZrC) composites are among the ideal materials for thrust vectoring control of missiles due to their erosion resistance and high temperature capability. Jet-vanes based thrust vectoring control requires in the initial phase of the launch where aerodynamic forces are insufficient to get required maneuverability. The environment experienced by the jet-vanes is very severe with typical gases temperature around 2500K at about 3-4 Mach of velocity; the exhaust gases contain hard tiny particles of alumina and results in severe erosion of jet-vanes. C/C composite blocks were fabricated by multiple impregnations of coal-tar pitch into 3D stitched carbon fibrous preform followed by carbonization/graphitization up to 2500oC. Carbon fiber reinforced zirconium carbide (C-C/ZrC) composites were fabricated by capillary infiltration of molten infiltration in a porous 3D stitched carbon-carbon (C/C) composite blocks. Zirconium infiltration was carried out under reduced pressure of 1x10-5 mbar and at 1900 and 2000 oC. The C/C-ZrC composites were characterized for mechanical and thermal properties. Flexural stress of the composites was determined as per ASTM C-1341 using 3-point bending fixture while tensile stress was measured using ASTM C-1273-15. Coefficient of thermal expansion (CTE) and thermal diffusivity were determined up to 1000oC in in-plane and through-thickness directions of the composite using 5x5x25mm specimens in a dilatometer. Thermal diffusivity of the composite samples was measured up to 1200oC as per ASTM E-1461-13 standard. Flexural and tensile strength of the composites are found to be varied between 125-180 MPa and 80-120 MPa respectively. CTE is found to be varied 0.5-2x10-6/oC (RT-1000oC) and 1.5-4x10-6/oC (RT-1000oC) in in-plane and through thickness directions respectively. Thermal diffusivity is found to be 45-15 mm2/s and 15-5 mm2/s (RT-1000oC) in the in-plane and through thickness directions. A typical size jet-vane (100x70x10mm) was exposed to the exhaust plume of a solid rocket motor and its ablation cum erosion resistance was determined. The eroded surface was characterized with XRD, XPS and morphology was studied using FESEM. Process details, properties, effect of infiltration temperature and the eroded surface morphology would be presented in this paper. In summary, the C/C-ZrC composite fabricated using the LMI route are found to have good

mechanical and thermal properties and demonstrated good erosion resistance to the plume of solid rocket motor exhaust. These composites can be used for applications like jet-vanes and nozzle throat.

Topic: 7 - Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)
Abstract no. 2089

# The sand-wind erosion behavior of C/SiC and its effect on ablation resistance

Presenting author: Dr CHENG Su
Affiliation: Northwestern Polytechnical University
youyi road? number 127,710072 Xi'an (Shaanxi, China)

Co-authors:

Carbon fiber-reinforced silicon carbide (C/SiC) composites with high temperature oxidation resistance and ablation resistance are significantly important candidates for thermal protection materials. The corrosion mechanism of C/SiC composite materials is the basis of their optimization and application. In this study, structural evolution of C/SiC composites fabricated by chemical vapor infiltration was investigated in the wind-sand storage environment. Furthermore, the effect of wind-sand on their ablation behaviors under oxyacetylene flame environment was studied. The ablation morphologies and physical erosion mechanisms of C/SiC composites were analyzed and discussed. The results indicated that SiC coating was damaged under wind-sand environment lead to the failure of C/SiC composites. The extent of physical injury in sand-wind was a lot, and it had obvious effect on the ablation behavior of the C/SiC composites, as well on the tensile strength and bending strength.

Topic: 7 - Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)
Abstract no. 2439

# Fracture behaviour improvement of ceramic based matrix composites via microstructure architecture (FMs method)

Presenting author: Prof GHASSEMI KAKROUDI Mahdi
Affiliation: University of Tabriz
29 bahman,51666 Tabriz (East Azarbaijan, Iran)

Co-authors: SHAHEDIFAR Vahideh (University of Tabriz), POURMAHMMADIE VAFA Naser (University of Tabriz), HASSANGHOLIZADEH M. (University of Tabriz)

There are many research to prevent the catastrophic fracture of ceramics and improve its fracture toughness. A novel method to make ceramic composites with a distinct fibrous structure, including of a major cell phase from a hard, strong material surrounded by a thin cell boundary of a weaker material was successfully developed. The resultant ceramics were called “fibrous monolithic ceramics (FMs)”. Non-brittle failures are frequently observed in FMs due to crack interactions with the weak cell boundaries as graphite. In other words, when the crack propagates thru the weak cell boundaries, a high energy absorption occurs because of tensile crack deflection or crack delamination. According to researchers' findings, crack interactions depend upon the properties of both the cell and cell boundaries, as well as the elastic mismatch between them, fracture resistance of the interface, and microcracks due to residual stress. Accordingly, in this research, a fibrous monolithic structure based on TaC-20 vol% HfC composite as cell (C) materials and porous graphite as cell boundary (CB) phase was produced by co-extrusion processing. Along with the fibrous composites, monolithic composites with the same composition of the cell was prepared. Then, both monolithic and fibrous composites were sintered using hot pressing at the temperature of 1800 °C for 1 h under a pressure of 40 MPa. The fracture behaviour of monolithic and fibrous specimens was compared and the fracture mechanisms were studied. Toughening mechanisms such as crack deflection and crack delamination were observed in the fracture microstructure of the hot pressed composites. By increasing the CB:C volume ratio in FM samples, the fracture toughness as well as work of fracture enhanced considerably. It was found that the fracture behaviour of the FM ceramics was severely dependent upon the proportion and the properties of the cell and cell boundary materials.

Topic: 7 - Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)
Abstract no. 2040

# SHS/RMI process for the synthesis of Ti3SiC2/SiC ceramic matrix composites from macrosized non-powder forms of titanium metal


Presenting author: Dr ISTOMIN Pavel

Affiliation: Institute of Chemistry, Komi Science Centre of the Ural Branch of the Russian Academy of Sciences
Pervomaiskaya, 48,167982 Syktyvkar (Russia)

Co-authors: ISTOMINA Elena (Institute of Chemistry Komi Science Centre of the Ural Branch of the Russian Academy of Sciences), NADUTKIN Aleksandr (Institute of Chemistry Komi Science Centre of the Ural Branch of the Russian Academy of Sciences), GRASS Vladislav (Institute of Chemistry Komi Science Centre of the Ural Branch of the Russian Academy of Sciences)



A novel approach for the synthesis of Ti3SiC2/SiC ceramic matrix composites using macrosized non-powder forms of titanium metal, such as foils, sheets, rods, tubes, etc., as a reactant have been developed. According to this method, dispersions of SiC particles in slurries or pastes as well as tape casted polymer films filled with SiC particles can be used as a second component of the reaction system. The reactants have to be assembled in a special manner and then heat treated under oxygen-free conditions at 1350–1500°C in order to enable self-propagating high-temperature synthesis (SHS) and reactive melt infiltration (RMI) processes to occur. This allows the reactive assemblies to be sintered into a dense ceramic composite with a damage tolerant Ti3SiC2-based matrix reinforced with hard SiC particles. It has been demonstrated that there is no need for a high pressure assistance during the sintering process. The proposed approach can be successfully applied for fabricating Ti3SiC2/SiC ceramic matrix composites with specified internal structures, including cellular and cannular ones. It is expected that the use of macrosized non-powder forms of titanium metal in the SHS/RMI synthesis of Ti3SiC2 MAX phase ceramic materials can provide them with important competitive advantage through an increase in their manufacturing productivity.


This work was financially supported by the Russian Foundation for Basic Research (grant #19-08-00131).

Topic: 7 - Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)
Abstract no. 2440

# Recent developments in ZrB2-based composites research in Iran


Presenting author: Prof SHAHEDI ASL Mahdi
Affiliation: University of Mohaghegh Ardabili
29 bahman,51666 Ardabil (Iran)

Co-authors: GHASSEMI KAKROUDI Mahdi (University of Tabriz), FARBAOD Foad (University of Tabriz), HASSANGHOLIZADEH M. (University of Tabriz)



As a member of ultrahigh temperature ceramics (UHTCs), zirconium diboride (ZrB2) has a collection of excellent characteristics including high melting point, hardness and elastic modulus, chemical inertness against molten metals as well as low thermal and electrical resistivity. Hence, it can be used in a variety of industrial applications such as aerospace vehicles, cutting tools, molten metal crucibles and electrodes. However, due to its covalent bonding, low self-diffusion coefficient and presence of oxide contaminations on the surface of this non-oxide ceramic, production of fully dense parts with acceptable properties is still a challenging issue.

Recently, many scientists and researchers around the world are investigating about the densification, microstructure, mechanical and thermal properties as well as oxidation behavior of ZrB2-based UHTCs. By searching for the word "ZrB2" in the Scopus database, there are over 1,400 documents published in the last 10 years. The Iranians, having published more than 80 scientific papers on this subject, are ranked 5th in the world after the countries of China, the United States, India and Japan. Most articles of Iran are published by researchers from University of Mohaghegh Ardabili, Materials and Energy Research Center, University of Tabriz and Iran University of Science and Technology, so that the authors of this article are at the forefront of them.

Iranians have generally employed pressureless sintering, hot pressing and spark plasma sintering to consolidate the ZrB2-based composites. They have incorporated many additives and reinforcements such as carbides, carbons, nitrides, oxides and metals to improve the sinterability, densification behavior, oxidation resistance and mechanical properties (e.g. flexural strength and fracture toughness) of this UHTC. The results of these research works have been published in the reputed international journals.

Topic: 7 - Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)
Abstract no. 2125

# Synthesis of High Purity Yttrium Diborocarbides Powder by High-Energy Ball-Milling and Reactive Spark Plasma


Presenting author: Dr NGUYEN Van-Quyet

Affiliation: Powder & Ceramics Division, Engineering Ceramics Department, Korea Institute of Materials Science
Changwon 51508, South Korea,51508 Changwon (South Korea)

Co-authors: LEE Sea-Hoon (Powder & Ceramics Division Engineering Ceramics Department Korea Institute of Materials Science), LEE Hee-Jung (Powder & Ceramics Division Engineering Ceramics Department Korea Institute of Materials Science), KIM Jun-Seop (Powder & Ceramics Division Engineering Ceramics Department Korea Institute of Materials Science)



In this study, high purity yttrium diborocarbides (YB2C2) powder was synthesized via modified spark plasma sintering (SPS) at 1500-1800°C for 60 min in a vacuum (?20 Pa) using Y2O3, B4C and carbon powder as starting materials. The effect of sintering temperature on the phase composition and microstructures of synthesized powders were characterized with x-ray diffraction (XRD), field-emission scanning electron microscopes (FE-SEM) and transmission electron microscopy (TEM), thermo-gravimetric analysis and differential thermal analysis (TGA-DTA). Fine and homogeneously intermixed raw powders were observed after high energy ball milling with nearly spherical morphology. The average particle size of the synthesized powder at optimum temperature (1600-1650?) was 300 nm. The metal basis purity of YB2C2 powder was 99.84%. Grain growth occurred strongly and the formation of layered structure became distinct when the synthesis temperature increased to 1800°C. X-ray pure YB2C2 specimen was obtained after SPS of the synthesized powder at 1900°C for 60 min. at a pressure of 80 MPa. The present report proposes a method for the fabrication of X-ray pure YB2C2 which is a new class of materials combining excellent properties such as damage tolerance, thermal shock resistance and high temperature stability.

Topic: 7 - Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)
Abstract no. 2051

# Chemical modification of refractory carbides in a gas atmosphere of SiO

Presenting author: Dr ISTOMINA Elena
Affiliation: Institute of Chemistry, Komi Science Centre of the Ural Branch of the Russian Academy of Sciences
Pervomaiskaya, 48,167982 Syktyvkar (Russia)
Co-authors: ISTOMIN Pavel (Institute of Chemistry of Komi SC UB RAS), ISTOMINA Elena (Institute of Chemistry of Komi SC UB RAS)

The refractory carbides of group 4 and 5 transition metals, such as TiC, ZrC and TaC, due to their very high melting points are of great interest for ultra-high temperature applications. It is expected that the additives of silicon-containing phases can improve their properties such as sinterability, fracture toughness, oxidation resistance, etc. It would be promising to grow silicide phases directly on the surfaces of the carbide particles prior to the sintering. For this purpose we propose a new approach consisting in siliciding the carbide powders with gaseous SiO at the high temperature.
In this work we have studied siliciding of the ZrC, TaC, (Ti,Zr)C, (Ti,Ta)C powders by this method. A special multiplate reactor assembled of shallow corundum crucibles was used in the experiments. The equimolar powder mixture of silicon and silicon dioxide was used as the reactive source of the SiO gas. The heat treatments of the samples were carried out in a vacuum electric furnace at 1400°C for 1 hour at the continuous pumping out of evolved gases. It was found that silicides, namely ZrSi, Ta5Si3 and TaSi2, grow on the surface of the particles of the corresponding carbides during the siliciding treatment. The as-prepared powders with different contents of the silicide phases were hot pressed under 25 MPa at 1700°C for 1 hour. The densification behaviour during hot pressing, phase composition and microstructure of the prepared samples were characterized.
This work was financially supported by the Russian Foundation for Basic Research (grant #19-08-00131).

Topic: 7 - Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nano-laminated Ternary Carbides and Nitrides (MAX Phases)
Abstract no. 1983

# Preparation and Properties of C/ZrC-SiC Composites Prepared by Precursor Infiltration and Pyrolysis


Presenting author: Dr LIU Wei
Affiliation: Research Institute of Aerospace Special Materials and Processing Technology
Yun Gang Bei Li 40,100074 Beijing (China)

Co-authors: SUN Tongchen (Research Institute of Aerospace Special Materials and Processing Technology), LIU Junpeng (Research Institute of Aerospace Special Materials and Processing Technology), YU Yi (Research Institute of Aerospace Special Materials and Processing Technology), PEI Yuchen (Research Institute of Aerospace Special Materials and Processing Technology)



The C/ZrC-SiC composites are prepared by precursor infiltration and pyrolysis (PIP) process. The effects of pyrolyzed process on the structure and property for the composites are studied. What's more, the ablation resistance is also evaluated. It is showed that the higher heated temperature resulted in performance degradation due to damaging the carbon fiber, while the lower heated temperature also resulted in performance degradation due to the higher oxygen content in the matrix. Only the moderate heated temperature can prepare the composites with high performance. The tensile strength is 183MPa at 1600?, while the linear ablating rate of composites is only 0.00088 mm/s after the Arc Heated Tunnel experiment at 2850K for 320s. Moreover, the produced ZrO2 and SiO2 are the main reason of good oxidation and ablation resistance for C/ZrC-SiC composites.

# Topic: 8

# Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 2039

# Critical Challenges to T/EBC Systems for CMCS


Presenting author: Prof LEVI Carlos
Affiliation: University of California, Santa Barbara
1361D Engineering II,Materials Department,93106-5050 Santa Barbara (California, United States)
Co-authors: POERSCHKE David (University of Minnesota), SUMMERS William (University of California Santa Barbara), ERICKS Andrew (University of California Santa Barbara), ZOK Frank (University of California Santa Barbara)



The recent implementation of SiC-based ceramic composites (CMCs) in aircraft propulsion represents a paradigm shift in gas turbine technology, arguably comparable to the introduction of single crystal superalloy airfoils ca. 1980. While the potential improvement in temperature capability and engine efficiency is unprecedented, there are a number of issues that limit the full exploitation of such potential. In addition to the longstanding concern for low temperature oxidative embrittlement and the limited temperature capability of current bond coats and matrices, the susceptibility of the protective SiO2 to volatilization in the combustion environment requires the use of layered environmental barrier coatings (EBCs) to achieve durability targets. Most EBC concepts, however, are based on silicates and are thus susceptible to degradation by molten silicate deposits generically known as CMAS originating from mineral debris ingested into engines with the intake air. A top coat layer may then be required to mitigate this problem. This presentation will discuss an integrated approach to understand the mechanistic foundation of the environmental degradation of T/EBCs by CMAS. The initial approach is based primarily on current developments in relevant thermodynamic databases, kinetic studies and micromechanic models that help elucidate the roles of the CMAS and EBC compositions.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 2136

# Microstructural Evolution of Yb2Si2O7 in High-Temperature High-Velocity Steam


Presenting author: Prof OPILA Elizabeth
Affiliation: University of Virginia
395 Mccormick Rd,22904 Charlottesville (Virginia, United States)

Co-authors: RIDLEY Mackenzie (University of Virginia)



$Yb_2Si_2O_7$ is a leading candidate for environmental barrier coatings (EBCs) for SiC-based ceramc matrix composites, having an excellent thermal expansion match with SiC and phase stability with changes in temperature. While $Yb_2Si_2O_7$ is more stable than thermally grown silica in high-temperature high-velocity steam, it is still susceptible to loss of $SiO_2$ as $Si(OH)_4(g)$. In this work, the microstructural evolution of $Yb_2Si_2O_7$ is studied as a function of exposure temperature, time, and steam velocity. Exposures are conducted in a steam jet furnace at temperatures between 1200 and 1400?C, 1 atmosphere steam, and gas velocities between approximately 25 and 200 m/s. It is observed that the surface microstructure is highly dependent on gas velocity even for constant temperature and time exposures. At the lowest gas velocities, a porous $Yb_2SiO_5$ surface layer forms with rapid kinetics. At intermediate gas velocities, the $Yb_2SiO_5$ surface layer densifies, slowing the $SiO_2$ depletion rate. At the highest gas velocities $Yb_2SiO_5$ is further depleted to porous $Yb_2O_3$. Relative rates and hypotheses for these varying $SiO_2$ depletion mechanisms are discussed. Finally, $SiO_2$ depletion mechanisms are mapped out in temperature, time, velocity space to further develop capabilities to predict thermochemical life of $Yb_2Si_2O_7$ EBCs.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 2170

# Reaction-bonded $Al_2O_3$ coatings for $Al_2O_3/Al_2O_3$ Ceramic Matrix Composites


Presenting author: Dr MECHNICH Peter
Affiliation: German Aerospace Center (DLR), Institute of Materials Research
Linder Höhe,51147 Cologne (Germany)

Co-authors: SCHAPER Nadine (German Aerospace Center (DLR), Institute of Materials Research)



Ceramic matrix composites based on Al2O3 fibers and porous Al2O3 matrices are being developed for various applications in harsh environments. Thermo-chemical degradation of Al2O3 is associated with hydroxylation in hot H2O-rich atmospheres and subsequent volatilization of Al-hydroxide species. This kind of degradation can effectively be reduced by recession-resistant environmental barrier coatings (EBC). A key issue for CMC is the relative low mechanical stability of the porous matrices, therefore CMC surfaces are prone to selective erosion in high-velocity gas streams, e.g. during coating by thermal spraying technologies. But also application of CMC without EBC may be limited due to low surface stability. Reaction-bonded Al2O3 coatings can effectively homogenize and stabilize CMC surfaces. Dispersions of Al2O3 and Al are deposited on the surface and fired in air where Al is oxidized and bonds together with Al2O3 to the CMC surface. Such coatings can be used as bond-coatings for thermally sprayed top coatings. On the other hand, stand-alone RBAO coatings promise a simple and cost-effective solution for CMC applications where outstanding H2O-stability, i.e. EBC functionality is less important than e.g. erosion resistance. A key to successful processing of RBAO coatings is the careful selection of raw materials and process parameters adapted to the respective CMC substrate.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 2358

# PVD-based Environmental Barrier Coatings for SiC-SiC CMCs: processing, microstructure and cyclic behavior

Presenting author: Prof SCHULZ Uwe
Affiliation: German Aerospace Center (DLR)
Linder Hoehe,51170 Cologne (Germany)

Co-authors: LEISNER Vito (German Aerospace Center (DLR), NARAPARAJU Ravisankar (German Aerospace Center (DLR)

Ceramic matrix composites (CMCs) are promising materials for components in the hot section of gas turbines and are already applied in the latest generation of aero-engines. They can withstand higher service temperatures than the currently used Ni-based superalloys. However, SiC-SiC CMCs possess an insufficient oxidation resistance under the presence of rapidly flowing water vapor as it is present in combustion atmospheres. They suffer from volatilization of silicon hydroxide which leads to severe surface recession. Therefore, environmental barrier coatings (EBCs) are mandatory that protect the underlying CMC. In this presentation, multilayer EBCs manufactured by PVD methods are introduced. Both SiC-based CMCs and model SiC-alloys were coated by magnetron sputtering processes with a first layer of an oxide-modified silicon as the bond coat, an intermediate layer of a rare-earth di-silicate, a mono-silicate layer for protection against water vapor recession, and a final top layer applied by either sputtering or EB-PVD for CMAS protection. The coating architecture was designed to minimize chemical interactions between different layers and to have a strain tolerant microstructure. Samples were tested up to 1250°C in air in a furnace cycle test, under severe thermal gradient conditions in a burner rig, and under flowing water vapour at isothermal conditions. The EBC system showed no spallation after up to 1000hrs of cyclic testing and retains stable interfaces between the layers. While the uncoated substrate suffered from severe degradation under flowing water vapor and showed rapid loss of the matrix material after only 1h of testing, the EBC considerably lowered the mass loss and provides a good protection of the CMC in this test. The evolution of the microstructure of the coating layers during testing, interfacial reactions, and phase formation will be additionally addressed.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 1933

# Mass transfer in multilayered EBCs consisting of Yb-silicates under oxygen potential gradients at high temperatures

Presenting author: Dr KITAOKA Satoshi
Affiliation: Japan Fine Ceramics Center
2-4-1 Mutsuno, Atsuta-ku,456-8587 Nagoya (Japan)

Co-authors: MATSUDAIRA Tsuneaki (Japan Fine Ceramics Center), WADA Masashi (Japan Fine Ceramics Center), YOKOI Taishi (Japan Fine Ceramics Center), YAMAGUCHI Norio (Japan Fine Ceramics Center), KAWASHIMA Naoki (Japan Fine Ceramics Center), OGAWA Takafumi (Japan Fine Ceramics Center), YOKOE Daisaku (Japan Fine Ceramics Center), KATO Takeharu (Japan Fine Ceramics Center)

Yb-silicates, especially $Yb_2SiO_5$ (YMS), have excellent water-vapor resistance, so that these materials are very promising as components of environmental barrier coatings (EBCs) for SiC fiber-reinforced SiC matrix composites (SiC/SiC). However, the thermal expansion coefficient of SiC/SiC is close to that of $Yb_2Si_2O_7$ (YDS), but smaller than that of YMS. Consequently, an EBC?typically has a composition gradient, with YDS and YMS applied as the bottom and top layers, respectively, which leads to a reduction of thermal stress during EBC fabrication and heat cycling. We have elucidated mass transfer processes within polycrystalline YDS and YMS monolayers based on evaluation of the oxygen permeability of wafers cut from sintered bodies as model EBCs. Oxygen permeation for both silicates was controlled by the inward diffusion of oxide ions and the outward diffusion of Yb ions. The mobility of silicon ions, which are the main constituent element of these oxides, was too small to contribute to the oxygen permeation. The oxygen shielding ability per unit grain boundary (GB) length of YMS was inferior to that of YDS. A dense YMS film was formed on a YDS wafer substrate using double electron beam physical vapor deposition (PVD) in conjunction with laser heating of the film as a multilayered model EBC. When the YMS film was subjected to a high oxygen partial pressure ($P_{O2}$(hi)) surface, the oxygen shielding ability was significantly enhanced by application of a large oxygen potential gradient ($d\mu_O$). The discontinuous increment of the Yb chemical potential ($\mu_{Yb}$) in a direction from the YDS substrate to the YMS film is probably responsible for the emergence of oxygen shielding ability.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 2282

# Environmental Barrier Coatings Made by Different Thermal Spray Technologies

Presenting author: Prof VAßEN Robert
Affiliation: Forschungszentrum Jülich GmbH, IEK-1
Leo Brandt Strasse,52425 Jülich (NRW, Germany)

Co-authors: BAKAN Emine (Forschungszentrum Jülich IEK-1), GATZEN Caren (Forschungszentrum Jülich IEK-1), KIM Seongwong (Forschungszentrum Jülich IEK-1), MACK Daniel Emil (Forschungszentrum Jülich IEK-1), GUILLONFORSCHUNGSZENTRUM JüLICH IEK-1 Olivier (Forschungszentrum Jülich IEK-1)

Environmental barrier coatings (EBCs) are essential to protect ceramic matrix composites against water vapor recession in typical gas turbine environments. Both oxide and non-oxide based CMCs need such coatings as they show only a limited stability. As the thermal expansion coefficients are quite different between the two CMCs, the suitable EBC materials for both applications are different. In the presentation examples of EBCs for both types of CMCs are given.
The most often used thermal spray techniques for the deposition of EBCs is atmospheric plasma spraying (APS). This technique with its major problems as limited crystallinity, crack formation or loss of constituents will be addressed. In addition, also results on more advanced thermal spray processes as high velocity oxygen fuel (HVOF), suspension plasma spraying (SPS) or very low pressure plasma spraying (VLPPS) will be described. Especially the last method appears suitable to deposit crystalline, dense coatings for example made of YB2Si2O7.
Finally, also results of the performance of the different coating systems with respect to thermal cycling, water vapor recession and partially CMAS attack will be presented.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 2047

# Improvement of mechanical properties by ytterbium silicide/SiC nanocomposites


Presenting author: Prof NAKAYAMA Tadachika
Affiliation: Nagaoka Univ of Tech
1603-1 Kamitomioka,940-2188 Nagaoka (Niigata, Japan)

Co-authors:



In this research, Yb2Si2O7-Yb2SiO5-SiC composite, a candidate for top coat materials of environmental barrier coating (EBC) for gas turbine engine blades, was synthesized. This composite was confirmed that having an excellent self-crack-healing ability, due to the combination of various morphology (whisker, particulate) of SiC nanofillers, and the volume expansion of Yb2Si2O7 when they are transformed from Yb2SiO5 at high temperature. However, once all the healing agents in the composite have reacted, the self-healing ability cannot be continued. In a real gas turbine system, the turbine blades are required to operate through many cycles and thousands hour. If the composite cannot regenerate the healing agent continuously, the self-healing property would be impractical. In order to retain this ability, in this research, we tried to regenerate Yb2SiO5 through a thermal process in water vapor. The experimental results showed that healing agent can be regenerated partly after the heat treatment. This opens a new door for the development of self-healing composite materials.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 1918

# Pyrochlore lattice tuning towards extremely low thermal conductivity for potential thermal barrier coating applications

Presenting author: Dr WANG YANFEI
Affiliation: National University of Defense Technology
109, Deya Road,410073 Changsha (Hunan, China)
Co-authors: LIU Rongjun (National University of Defense Technology)

Thermal barrier coatings (TBCs) play a crucial part in gas turbine engines to improve their operation temperatures and hence their thrust-to-weight ratio. A low thermal conductivity is a prerequisite for a TBC topcoat material. A2B2O7-based pyrochlores are proposed as a promising next-generation TBC material due to a combination of properties favorable for TBC applications. Above all, the crystal structure of pyrochlores is open and thus can be tuned by strategies such as doping. In order to achieve extremely low thermal conductivity throughout the whole temperature range, in this study, we present a comprehensive research on pyrochlore lattice tuning by doping both A- and B- sites. The results show that, by doping smaller cations on A-sites, a resonant phonon scattering effect can be generated and results in a dramatically reduced thermal conductivity throughout the whole temperature range. In addition, the doping of B-sites by a bigger cation tends to soften the pyrochlore lattice, which is beneficial to reduce high temperature plateau thermal conductivity. The combination of both A-site doping by a smaller cation, resulting in a rattling effect, and B-site doping by a bigger cation, resulting in a softened lattice, can potentially yield an extremely low thermal conductivity throughout the whole temperature range, which sheds some light on the selection of next-generation TBC material.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 2233

# Effect of difference in material of substrate on aerosol deposited mullite coating under heat exposure

Presenting author: Dr HASEGAWA Makoto
Affiliation: Yokohama National University
79-5, Tokiwadai, Hodogaya-ku,2408501 Yokohama (Kanagawa, Japan)

Co-authors: SHIBUYA Toshiki (Yokohama National University), IUCHI Atsuhisa (Yokohama National University)

Environmental barrier coatings (EBCs) are essential for SiC fiber reinforced SiC matrix composites to protect from high temperature oxygen and water vapor. Typical EBCs are composed of several ceramics layers formed by air plasma spraying (APS). However, the coating processed by APS has pores and amorphous phases. Due to the shrinkage of the coatings by sintering and crystallization at high temperature, vertical cracks initiate at EBCs. This promotes substrate oxidation and leads the coating spallation. In order to prevent them, the coatings are required to be dense from the as-deposit state. Aerosol deposition (AD) method is known to fabricate a dense and crystalline ceramic coating at room temperature. In this study, mullite powders are deposited on silicon and SiAlON substrates. Microstructural change of coatings during heat exposure in an air is investigated.

After deposition of mullite coating on a substrate for 15 µm in thickness, the deposited material was heat exposed at 1573 K. In the mullite coating on the silicon substrate, aluminum and oxygen mutually diffused during heat exposure. The mullite decomposed at the vicinity of the interface between mullite and silicon. On the other hand, decomposition of mullite coating was not observed on the SiAlON substrates in the same heat exposure condition. The debonding of the mullite coating on the SiAlON substrate was observed. There are two possible reasons for this debonding; thermal stress generated in the specimen during the temperature increase, and generation of nitrogen due to oxidation of SiAlON in an air. When the material was exposed in nitrogen gas, the debonding was not observed. Therefore, this debonding was occurred by the oxidation of SiAlON. When the mullite was deposited to 30 µm in thickness, mullite coating was maintained with no decomposition even after 100 hours heat exposure at 1573 K.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 2074

# Toward the understanding of failure mechanisms of environmental barrier coatings

Presenting author: Mme FERNANDEZ Manon
Affiliation: Laboratoire des Composites Thermostructuraux
3 Allée De La Boétie,33600 Pessac (France)

Co-authors: REBILLAT Francis (Laboratoire des Composites Thermostructuraux), COUEGNAT Guillaume (Laboratoire des Composites Thermostructuraux)

In the aeronautic field, current issues are the reduction of noise pollution, consumption and emissions of NOx and CO2. In this context, structures weight and operating temperature are key parameters. Among the candidate materials for such applications, SiC based ceramic matrix composites (CMC) have been selected.
To increase composites lifetime, protective coatings based on rare earth silicate have been developed. Their purpose is to limit the surface degradation by oxidation/corrosion of the pieces. Understanding the degradation and ultimately failure mechanisms of EBCs is crucial for the development of CMC technology. Two properties are essential for the environmental barrier to fulfil its protective role: adhesion to the substrate and thermomechanical and chemical stability of the coating.
A loss of adhesion over a long distance can lead to spalling of the EBC. This is a first failure mechanism, caused by the weakness of the interfacial bond between the coating and the substrate. This scenario of ruin seems mainly related to a phenomenon of growth of an unstable and cracked oxide scale leading to the creation of discontinuities at the EBC/CMC interface. With this in mind, work aimed at characterizing the morphology of the interface and quantifying adhesion degradation with aging under representative engine environment have been undertaken.
In addition to adhesion problems, the coating itself may also have weaknesses, detrimental to the lifetime of the system. A loss of EBC protection can occur following a surface recession due to the volatilization of the coating; quantification of the recession resistance of the EBC will be undertaken.
To better understand the importance of the various failure mechanisms determined experimentally, a modeling of the residual mechanical stresses developed in the EBC/CMC system has been proposed. The objective is to simulate and analyze the level of thermal residual stresses developed in each constituent of the system.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 1900

# Development of environmental barrier coatings for non-oxide ceramic matrix composites

Presenting author: M KLEMM Hagen
Affiliation: Fraunhofer IKTS Dresden
Winterbergstr. 28,01277 Dresden (Germany)

Co-authors: KUNZ Willy (Fraunhofer IKTS Dresden), SCHöNFELD Katrin (Fraunhofer IKTS Dresden), GRONDE Bernd (Fraunhofer IKTS Dresden)

Ceramic matrix composites (CMC) offer a high potential for applications as structural parts in advanced gas turbines. During recent years, significant progress in material development of oxide and non-oxide CMC has been achieved, however, there are still considerable deficits especially in the long-term behavior of the materials in hot gas conditions.
The present study is focused on the environmental stability of the materials. Caused by the high water vapor pressure in combination with high temperatures and gas velocities, corrosion processes at the surface and inside the materials were observed resulting in significant material degradation and mass loss. Hence, environmental barrier coatings (EBC) have been presented to be the solution to protect the surface of the ceramic materials.
Systematic studies on the hot gas corrosion of non-oxide CMC have been performed with and without EBC. Based on a detailed understanding of the processes in the whole system, EBC and the ceramic base material during application in hot gas environments at elevated temperatures, general concepts for the development of environmental barrier coatings will be discussed.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 2253

# Interfacial toughness of environmental barrier coatings (EBCs) with Si bond coat after heat exposure

Presenting author: Dr KAKISAWA Hideki
Affiliation: National Institute for Materials Science
1-2-1, Sengen,305-0047 Tsukuba (Japan)

Co-authors:

We investigated the change of interfacial toughness of environmental barrier coatings (EBCs) with Si bond coat produced by either of atmospheric plasma spray (APS) or reduced pressure plasma spray (LPPS) using a recently developed interface fracture test reproducing a low phase angle. Both Si layer produced by APS and LPPS had good adhesion with the substrate and upper oxide ceramic layer as coated. After the heat exposure in air, the interfacial toughness of EBCs with Si bond coat by APS decreased because a network of thin oxide layer was formed at the splat structure boundaries within the Si layer. We carried out theheat exposure for the EBCs in Ar gas atmosphere to investigate the origin of oxygen for the oxide network in the Si layer. The same experiments were also done for the EBCs with Si bond coat by LPPS. Based on the obtained results, the effect of plasma spray process on the formation of the thin oxide layer which caused the degradation of interfacial toughness of EBCs with APS Si bond coat will be discussed in the paper.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 2004

# Advanced oxide strengthened silicon bond coats with rare-earth silicate based EBCs for SiC/SiC CMCs


Presenting author: M LEISNER Vito
Affiliation: German Aerospace Center - DLR
Institute of Materials Research,Linder Höhe,51147 Cologne (Germany)

Co-authors: ANTON Ronja (German Aerospace Center - DLR), SCHULZ Uwe (German Aerospace Center - DLR)



The introduction of SiC/SiC CMCs in aero engines demands for well-tailored Environmental Barrier Coatings (EBC). Physical vapour deposition (PVD) coating technology was employed to manufacture these systems since this technology is potentially favourable for the application on near net shape designed CMC components. They have further advantages such as distinct control over microstructure and tailored chemistry. Magnetron sputtered three-layer EBC systems that consist of a silicon-based bond coat (BC), a rare-earth di-silicate intermediate layer and a rare-earth mono-silicate top coat against water vapour attack have been developed and continuously optimised by our group. Thermal sprayed pure silicon is the state-of-the-art choice for the BC. In this talk, not only the performance of the PVD counterpart, but also novel variants with advanced properties will be presented. The longevity and thermomechanical properties of the BC have been enhanced by oxide strengthening with yttria or hafnia dopants. Both dopants evoke significant differences in the morphology of the coatings. Hafnia formed homogeneously distributed particles that form a microscale network within the silicon matrix and was later transformed into hafnon. Contrary, yttria formed larger yttrium-silicates and pores that were closed with time. Four variants were tested and showed improved resistance against spallation and cracking compared to pure silicon after thermal cyclic oxidation testing at 1250°C for 1000hrs. Some variants exhibited faster oxidation kinetics, some were still in the range of the slow silicon oxidation. 3D-reconstruction and TEM analyses gave insight into the microstructural features of the hafnia doped BCs. They revealed the underlying mechanism of oxidation and hafnon formation. The compatibility of rare-earth silicates with the novel bond coats have been validated with both furnace cycle testing and isothermal testing in streaming water vapour at 1250°C. The results proofed their excellent functionality as alternative bond coats for EBCs.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 2087

# Yb-Silicate Environmental Barrier Coatings for SiC/SiC CMCs


Presenting author: Dr BAKAN Emine
Affiliation: Forschungszentrum Jülich GmbH, IEK1
Wilhelm-Johnen-Straße,52428 Jülich (Germany)

Co-authors: MACK Daniel (Forschungszentrum Jülich GmbH IEK-1 52425 Jülich Germany), KOCH Dietmar (German Aerospace Center (DLR), Institute of Structures and Design 70569 Stuttgart Germany), KLEMM Hagen (Fraunhofer Institute for Ceramic Technologies and Systems IKTS Dresden 1277 Dresden Germany), VAßEN Robert (Forschungszentrum Jülich GmbH IEK-1 52425 Jülich Germany)



Environmental barrier coatings (EBCs) are needed to protect SiC structures exposed to high temperatures in water vapor-rich environments. The state of the art EBC system for SiC/SiC CMCs is consisting of a silicon (Si) bond coat and ytterbium disilicate (Yb2Si2O7) volatilization top coat barrier. The material of choice for environmental stability is ytterbium monosilicate (Yb2SiO5) however it has a higher coefficient of expansion compared to the CMC substrate at the same time. Yb2SiO5 is also an unavoidable secondary phase in the Yb2Si2O7 coatings when conventional thermal spray methods are used for deposition. Significant amount of Yb2SiO5 results in through thickness vertical cracking in the coatings and thereby accelerated degradation. In this paper, strategies for avoiding the vertical cracking in the Yb2Si2O7 – Yb2SiO5 layers will be discussed. Effect of low/high Yb2SiO5 phase content in Yb2Si2O7 coatings on environmental stability will be demonstrated by high-velocity water vapor (v=100m/s, PH2O=0.15 atm) recession test results of the coatings. Finally, thermal gradient cycling test results of conventional and advanced processed Si/ Yb2Si2O7 EBCs in the modified burner rig set-ups of JÜLICH will be presented. Effect of surface and substrate temperatures in relation to degradation mechanisms of coatings will be examined.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 2295

# Effect of EBC porosity on performance at 1300°-1425°C in steam

Presenting author: Dr PINT Bruce
Affiliation: Oak Ridge National Laboratory
PO Box 2008,37831 Oak Ridge (TN, United States)

Co-authors:

The next generation of ceramic matrix composites may target operation above 1400°C, therefore, an environmental barrier coating (EBC) without a Si bond coating may be needed. To begin to understand EBC performance under those conditions, initial experimental work is evaluating the cyclic oxidation performance of two ytterbium silicate coatings with different levels of porosity on CVD SiC substrates at 1425°C in flowing steam. In addition, similar specimens were cycled in air+90%H2O at 1300°C for 500 1-h cycles with and without a Si bond coating. The two porosity levels did not show a difference in performance. However, without a pre-anneal at 1300°C, the EBC had poor adhesion without a bond coating and spalled within 20 cycles at 1300°C. With annealing prior to exposure, the coatings all survived for 500 cycles and their reaction products are being characterized. Research sponsored by the U. S. Department of Energy, Office of Fossil Energy, Advanced Turbine Program.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 1857

# Development of Environmental Barrier Coatings for SiC/SiC Ceramic Matrix Composites via CVD


Presenting author: M KöNIG Till
Affiliation: DECHEMA-Forschungsinstitut
Theodor-Heuss-Allee 25,60486 Frankfurt am Main (Germany)

Co-authors: UHLMANN Franziska (MTU Aero Engines Dachauer Str. 665 80995 München Germany), HILSER Ludwig (MTU Aero Engines Dachauer Str. 665 80995 München Germany), GALETZ Mathias (DECHEMA-Forschungsinstitut Theodor-Heuss-Allee 25 60326 Frankfurt am Main Germany)



With increasing combustion temperature of turbine engines the commonly used nickel-based superalloys are getting closer to their melting point and excessive cooling along with thermal barrier coatings are used to maintain the properties at the desired level. The substitution of these alloys by Ceramic Matrix Composites (CMC) for applications in the high temperature region of turbines is widely discussed and first parts are in service. Due to their mechanical properties at high temperatures especially SiC/SiC-CMC are investigated. The efficiency of the turbines can be increased by using higher combustion temperatures, the abandonment of cooling and reducing the mass. In oxidizing atmospheres these materials form silica scales, which are generally considered protective. But the presence of water vapor results in the volatilization of the silica scale by the formation of hydroxides. The simultaneous process of oxidation and evaporation makes it necessary to apply coatings that reduce the evaporation and result at best in parabolic kinetics.
Instead of the commonly used thermal spraying the application of environmental barrier coatings via the chemical vapor deposition process (CVD) is investigated. Potential advantages of the applied process are total coverage even in undercuts and a chemical bonding to the substrate. Two different coating systems were investigated, both with the aim to grow aluminum oxide at the operating conditions. In the first approach a two-step pack cementation process was used to apply layered coating. The second approach was to apply an aluminum forming coating by the direct deposition via a high temperature CVD process. The oxidation of these coatings was investigated in water vapor containing atmospheres in comparison to the uncoated material. Thermal cycling and 4-point-bending-tests were used to investigate the adherence. The microstructure of the coatings was examined before and after the exposure using X-ray diffraction, scanning electron microscopy and electron beam microanalysis.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 2050

# Corrosive Properties of RE-doped Silicate for Environmental Barrier Coatings

Presenting author: Prof JANG Byung-Koog Jang
Affiliation: Interdisciplinary Graduate School of Engineering Sciences, Kyushu University
6-1 Kasuga-koen,816-8580 Kasuga-shi (Fukuoka, Japan)

Co-authors: LEE Kee-Sung (Kookmin University), KIM Seongwon (Korea Institute of Ceramic Engineering and Technology), OH Yoon-Suk (Korea Institute of Ceramic Engineering and Technology), LEE Sung-Min (Korea Institute of Ceramic Engineering and Technology), KIM Hyung-Tae (Korea Institute of Ceramic Engineering and Technology)

The improvement of the gas turbine inlet temperature is a key factor involved in increasing the fuel efficiency and reducing the carbon emissions of a gas turbine. Due to the high limit point of temperature capability, non-oxide silicon-based ceramics, such as SiC/SiCf, Si3N4 and SiC, have been investigated extensively as potential structural material for hot gas parts for next-generation gas turbines. However, abundant investigations have indicated that the disadvantageous factor of Si3N4 and SiC as it applies to gas turbines is that they lose observable weight in the combustion environment.

In the present study, the influence of isothermal heat treatment on thermo-chemical properties of Y2SiO5 EBCs (environmental barrier coatings) on SiC was investigated. The hot corrosion between Y2SiO5 coatings and artificial CMAS (CaO–MgO–Al2O3–SiO2) was examined by isothermal heating at 1400?C in air during 1~50 hrs. The evaluation of hardness and Young’s modulus was performed on the cross-section of Y2SiO5 coatings by nano indentation method. The isothermal heat treatment improves the hardness and Young’s modulus of Y2SiO5 coatings. In addition, high-temperature corrosion behavior of volcanic ash and CMAS on sintered Yb2SiO5 are investigated.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 1829

# Enhanced EBC Development and Behaviour Analysis for High Temperature CMC Components


Presenting author: Dr PIN Lisa
Affiliation: SAFRAN Ceramics
105 avenue Marcel Dassault,33700 Mérignac (France)

Co-authors: BOUILLON Eric (SAFRAN Ceramics), ARNAL Simon (SAFRAN Ceramics)



Silicon carbide fiber reinforced silicon carbide matrix composites (SiC/SiC CMC's) are attractive materials for use in gas turbine hot sections due to the potential for high temperature mechanical properties and overall lower density than metals. However silicon carbide matrix composite are damaged under high temperature combustion environments: development of thermally grown oxide and volatilisation of silica under water vapour at high temperature. This results in unacceptable recession of the surface. That is why, it is necessary to develop an environmental barrier coating to prevent accelerated oxidation by limiting oxidant access to the surface of the silica former. This coating requires many criteria in order to be used as an environmental barrier coating : low oxygen permeability, coefficient of thermal expansion close to that of the silicon carbide matrix composites to prevent delamination or cracking, mechanically and chemically stable under thermal exposure.

This paper proposes to present a report on environmental barrier coating investigations in SAFRAN, and more particular on the diffusion mechanisms of oxidizing species depending the composition and the microstructure of the environmental barrier coating.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 2001

# Thermal properties and crystal structure of ytterbium titanate

Presenting author: Dr TANAKA MAKOTO
Affiliation: Japan Fine Ceramics Center
2-4-1 Mutsuno, Atsuta-ku,4568587 Nagoya (Japan)

Co-authors: ASAI KENTA (Gifu University), OGAWA TAKAFUMI (Japan Fine Ceramics Center), KAWASHIMA NAOKI (Japan Fine Ceramics Center), KITAOKA SATOSHI (Japan Fine Ceramics Center), IZUMI FUJIO (Japan Fine Ceramics Center), YOSHIDA MICHIYUKI (Gifu University), SAKURADA OSAMU (Gifu University)

Thermal and environmental barrier coatings (T/EBCs) with exceptional heat cycle resistance and environmental shielding play a key role in allowing SiC/SiC composite to be applied to advanced high-pressure turbine components. T/EBCs typically incorporate a multilayered structure, with the aim of obtaining exceptional performance through the use of layers with individual characteristics. In general, when an oxide with low thermal conductivity that is essential to enhance heat insulating performance is applied as the top layer of T/EBCs, the surface temperature tends to be increased. This results in water vapor volatilization and/or CMAS degradation of the constituent layers, leading to collapse of the multilayered structure. Contribution of thermal radiation energy becomes larger as the temperature increases. Oxide materials constituting T/EBCs and CMAS have transparency to this radiation. Therefore, if thermal radiation energy could be effectively reflected from a T/EBC, then the resulting decrease in the temperature of the top surface would mitigate the CMAS attack. We have considered the use of thermally reflective T/EBCs that consist of two different oxide materials with a large difference in refractive index (n). Combinations of Yb2Ti2O7 (high n)/Yb2Si2O7 (low n) and Yb2TiO5 (high n)/Yb2SiO5 (low n) are expected to be potential candidates that also exhibit excellent corrosion resistance. Furthermore, Yb2Ti2O7 has lower thermal conductivity than conventional yttria-stabilized zirconia. The pyrochlore Yb2Ti2O7 is transformed to the fluorite Yb2TiO5 with an increase in the content of Ti in the Yb2+xTi2-xO7-x/2 series; however, the details of the phase boundary and the crystal structure of the solid solution have yet to be clarified. In this study, the thermal conductivities of Yb2+xTi2-xO7-x/2 were evaluated at high temperatures, and the relationship between the composition and thermal conductivity is discussed with respect to the change in the crystal structure analyzed by Rietveld method and its applicability.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 1955

# CMAS degradation of environmental barrier coatings in presence of water vapor and air

Presenting author: Mme SCHöNFELD Katrin
Affiliation: Fraunhofer IKTS
Winterbergstrasse 28,01277 Dresden (Germany)

Co-authors: KLEMM Hagen (Fraunhofer IKTS)

Environmental barrier coatings (EBC) are increasingly susceptible to degradation by molten calcium-magnesium alumino silicate (CMAS) deposits in advanced engines that operate at higher temperatures. When the siliceous minerals deposit on the surface of EBCs, a glassy melt of calcium–magnesium–aluminosilicate (CMAS) will be formed, leading to the EBCs degradation.
In this investigation the most common CMAS composition 33CaO-9MgO-13Al2O3-45SiO2 was used as deposition on top of suitable EBC materials. The prepared materials were stored at 1200 ° C under the influence of water vapor or air to analyze the corrosion behavior.
The results indicated that the penetration of molten CMAS was very different for the investigated materials. For the oxidic materials ZrO2 und YAG the penetration and reaction of CMAS was less compared to the rare-earth silicates (Y2SiO5, Yb2SiO5 and Yb2Si2O7). Chemical reactions between the CMAS and the base material were observed in the silicate materials, with formation of barrier layers in the monosilicates, which limited the further degradation. For the disilikate the reaction zone was dissolved into molten CMAS and no barrier layer was formed. In this case, the hot gas corrosion was most pronounced, since the reaction of SiO2 with water vapor to volatile hydroxides could be carried out without inhibition.
Finally, investigations on SiC / SiC composites with the typical coating system Si Bond coat and Yb2SiO5 or Yb2Si2O7 Top coat were carried out. For the coating the APS method was used. The corrosion behavior under the influence of CMAS, which was previously analyzed on the monolithic ceramics, was confirmed at the APS coatings. The corrosion rate of the Yb2Si2O7 layer was significantly higher than that of the Y2SiO5 layer.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 2174

# Thermal fatigue cracks on environmental barrier coating under thermal gradients

Presenting author: M ARCHER Thibaut
Affiliation: ONERA
Avenue de la division Leclerc,92230 Chatillon (France)

Co-authors: BEAUCHêNE Pierre (ONERA), HUCHETTE Cédric (ONERA), LACOMBE Benjamin (Safran Ceramics), VANDELLOS Thomas (Safran Ceramics), HILD François (Laboratoire Mécanique et Technologie (LMT), Cachan)

Because of their high temperature mechanical and physical properties and their low density, ceramic matrix composites are currently investigated for their use in the hot sections of future turbine engines. However, for these applications, the presence of water vapor leads to surface recession of the protective silica layer formed at high temperatures. It is therefore essential to protect CMCs not only to withstand operating temperatures but also corrosive environments, hence the development of new thermal and environmental barrier coatings (T/EBCs).
In turbine engines, components will face hot gas steam and forced air cooling, generating multiaxial thermal gradients but also mechanical loadings. These conditions make standard tests used for characterization ill-adapted to assess damage initiation and growth or estimate lifetime. For that reason, the coated CMC is locally heated using a high power (3kW) high heat flux CO2 laser to perform thermal fatigue tests.
To understand the behavior of the material, the front (i.e. coated) side is monitored using a middle-wave infrared camera to measure and reconstruct temperature fields considering the change of emissivity of the coating as a function of temperature, an optical camera for the use of digital image correlation up to very high temperatures thanks to a speckle pattern created using SiC powders and optical filters. The through-thickness thermal gradient is controlled by active air-cooling at the back side of the sample and bichromatic pyrometers. Damage onset and growth is detected in-situ using acoustic emission with wire waveguides. After the test, non-destructive evaluations using laser spot thermography show the development of thermal fatigue cracks located under the laser beam, SEM observations confirm these results.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 1884

# Advances in the electrophoretic deposition of ceramics:example of rare-earth silicate coatings


Presenting author: Mme PRIOUX Manon
Affiliation: CIRIMAT
bâtiment CIRIMAT,118 route de Narbonne,31062 Toulouse cedex 9 (Haute-garonne, France)
Co-authors: ANSART Florence (CIRIMAT), DULUARD Sandrine (CIRIMAT), PIN Lisa (SAFRAN CERAMICS), ARNAL Simon (SAFRAN CERAMICS), PUJOL Guillaume (DGA), GOMEZ Philippe (DGA)



ElectroPhoretic Deposition (EPD) relies on the migration of charged species, i.e. dispersed particles in a liquid medium, under the effect of an electrical field. It is a process easy to implement and suitable for the deposition and shaping of different nature of coatings, even on complex shapes. However, the mechanisms leading to electrophoretic deposition are complex and a rigorous control of many parameters is required. The first empiric law proposed of the EPD process was proposed by Hamaker in 1910. The Hamaker's law states that the amount of powder brought to the electrode depends on the electrophoretic mobility of the powder in suspension, the electrical field strength between the electrodes, the solid loading and the surface area of the substrate, and evolves linearly with time. These parameters are related either to the deposition medium or to the process parameters, and are both considered in this study. A preliminary formulation work on solid content, additives, solvent composition has been carried out. Then EPD intrinsic parameters have also been optimized for the formation of homogeneous and reproducible coatings. The influence of the roughness and the resistivity of the substrate on the adherence and the morphology of the coating have also been studied. This work highlights the influence of these various parameters in the case of rare earth silicates based coatings, and compares experimental values and theoretical ones. One of the objectives is to optimize the suspension chemistry in order to obtain dispersion suitable for the formation of compact and protective ceramics coatings.  The experiments were carried out in organic liquid medium such as ethanol and isopropanol. The use of currents in the range of 25 and 150 $\mu A/cm^2$ leads to homogeneous and conformal coatings. These experiments allow to establish correlations between EPD parameters and coatings microstructure and morphology.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 2072

# Development of an advanced bond coat for environmental barrier coating


Presenting author: Mme SALLES Marie
Affiliation: Laboratoire des Composites ThermoStructuraux
3 allée de la Boétie,33600 Pessac (France)

Co-authors: REBILLAT Francis (Université de Bordeaux LCTS), LE PETITCORPS Yann (Université de Bordeaux LCTS)



Environmental barrier coatings (EBCs) are under development to protect SiC/SiC ceramic matrix composites (CMCs) used in hot-section components of gas turbine engines. To lower emissions and to increase fuel efficiency, new EBC materials able to withstand higher combustion temperatures are required. One of the most reported failure mode of such a protection is spallation due to oxidation by water vapor. During operation, water vapor diffuses through the top coat material (rare earth disilicate) and reacts with the silicon bond coat to form a SiO2 layer known as TGO (Thermally Grown Oxide). The formation of this oxide layer generates multiple stresses limiting the EBC life: growth stress caused by the x2.2 volume expansion between silicon and silica, phase transformation stress due to ? to ? cristobalite transition during cooling causing a 5% volume contraction, and thermal stress because of the thermal expansion mismatch between the constituants (CMC/Si/cristobalite/EBC).
This work aims to identify new bond coat compositions to enhance EBC lifetime. Additions such as boron are investigated to explore their effect on oxidation kinetic and silica phase transformation. Samples are prepared by Spark Plasma Sintering and exposed to oxidation at 1300°C for 100 hours in a moist oxidizing environment. Weight changes during oxidation, TGO thickness, nature and structure of the oxide(s) formed are compared with the current silicon bond coat system. Kinetic laws of oxidation are extracted by taking into account the simultaneous formation and volatilization of the silica scale.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 2014

# ADVANCED ENVIRONMENTAL BARRIER COATINGS FOR AL2O3/AL2O3 CMCS MANUFACTURED BY THERMAL SPRAY PROCESSES


Presenting author: Dr MACK Daniel Emil
Affiliation: Forschungszentrum Jülich HmbH
Wilhelm-Johnen-Str,52425 Jülich (Germany)

Co-authors: MACK Daniel Emil (Forschungszentrum Jülich GmbH), GUILLON Olivier (Forschungszentrum Jülich GmbH), VAßEN Robert (Forschungszentrum Jülich GmbH)



Al2O3/Al2O3 ceramic matrix composites (CMC) are candidate materials for high-temperature applications such as gas turbines. As water vapor corrosion of oxide/oxide CMC is a major issue, the application of suitable environmental barrier coatings (EBC) is inevitable for long time use. Current state-of-the-art EBCs consist of Y2O3 which can be easily processed in atmospheric plasma spraying (APS), whereas the utilization of materials with further improved corrosion resistance such as YAlO3 or ZrO2 is hindered by phase instabilities during manufacture or the thermal expansion mismatch.

In this study, advanced processing technologies as very-low pressure plasma spraying (VLPPS) and laser surface texturing (LST) have been assessed to overcome these obstacles. The suitability of laser ablation for surface preparation of Al2O3/Al2O3 CMCs was examined and laser parameters were optimized to create interface structures like for example a honeycomb or a cauliflower like structure. Materials including Y2O3, YAlO3, YSZ and Gd2Zr2O7 were deposited on non-structured and structured CMC substrates by APS and VLPPS. Microstructures and chemical stability of the coatings were analyzed and the performance was tested in terms of adhesion strength as well as in terms of degradation under high-temperature exposure including furnace cycling and burner rig tests. Results indicate that the enhanced processing enables the utilization of the new materials.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 2005

# Damage characterization of EBC-SiC/SiC Ceramic Matrix Composites under complex thermomechanical loading paths


Presenting author: Dr LEGIN Blanche
Affiliation: Institut Pprime, CNRS – ISAE ENSMA – Université de Poitiers – UPR 3346 DPMM, ENSMA – Téléport 2,1 avenue Clément Ader,86961 Futuroscope-Chasseneuil Cedex (France)
Co-authors: MAUGET Florent (Institut Pprime), CORMIER Jonathan (Institut Pprime), PANNIER Yannick (Institut Pprime), GRANDIDIER Jean-Claude (Institut Pprime), REVEL Thomas (Safran Ceramics)



SiC/SiC ceramic matrix composites (CMC) are promising materials for high temperature applications. CMCs are used as structural materials in hot sections of aero-engines, and they are therefore submitted to harsh environmental service conditions in addition to mechanical loading that may cause development of damage. To prevent environmental degradation, CMCs are protected by an environmental barrier coating (EBC). This coating integrity must then be maintained during the expected lifetime of the parts. A perfect understanding of CMC/EBC system is needed to allow the introduction of CMCs/EBCs system in next generation engines.
The work herein presented aim to investigate the damage mechanisms of EBC-SiC/SiC ceramic matrix composites during thermomechanical fatigue (TMF) loading and under corrosive environment that are similar to service conditions.
Experimental tests are performed using the MAATRE burner rig which is available at Institut Pprime. This test rig combines a gas burner with a mechanical testing machine so test structures are simultaneously mechanically loaded and exposed to oxidizing gas stream at very high temperatures. For this work, specimens have been studied under TMF. Temperature in the test section varies between 250°C and 1600°C and a constant load is applied during cycling. Different cooling rate conditions have been evaluated.
Several experimental tools are used to characterize thermal gradient and investigate damage occurring during cycling. A typical cracking pattern is observed among damages on the outer surface of the coating.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 2070

# Self-healing behavior of Mullite-based ceramics for environmental barrier coatings


Presenting author: Prof LEE Kee Sung
Affiliation: Kookmin University
Mechanical Eng. Kookmin Univ.,77, Jeongneung-ro, Seongbuk-gu,02707 Seoul (Seoul, South Korea)
Co-authors: SEO Hyoung-IL (Kookmin University), KIM Dae Jong (Korea Atomic Energy Research Institute)



A gas turbine hot gas component operating at temperatures more than 1500oC requires heat resistant fiber reinforced CMC. The SiCf/SiC CMC is also expected to be able to withstand high temperature fatigue and creep. However, the environmental barrier coatings (EBCs) are indispensable for protecting the SiCf/SiC CMC from oxidation and water vapour penetration. The cracking and delamination of coating layer is one of the problems in multiple heat cycles of turbine system, where heating and cooling are repeated periodically, and especially the abrupt stopping of system causes the formation of cracks.

In this study we fabricated Mullite and Mullite+Yb2SiO5 coating by plasma spray on Si bondcoat on the SiCf/SiC CMC. The CMC was fabricated by LMI method. Thermal shock cycles were performed up to 5,000cycles from high temperature of 1350oC to air to induce the cracks in the coating layer. Typical mud-cracks on the surface and unidirectional vertical cracks on the section were observed by thermal shock.

The crack healing phenomena were observed in the Mullite-based ceramics from 3000 cycles of thermal shock by melting, filling and crystallization of new phase. The crack density was diminished according to thermal shock cycles. Post indentation after thermal shock cycle indicates that mechanical degradation is not much degraded in the Mullite+ Yb2SiO5 coating.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 2265

# Interplay between phase stability, segregation and local stresses in nanocrystalline entropy stabilized transition metal oxides


Presenting author: M NALLATHAMBI Varatharaja
Affiliation: Research Scholar
Room no:1003, Mahanadhi hostel,IIT Madras,600036 Chennai (Tamil Nadu, India)
Co-authors: BHASKAR Lalith Kumar (Research Scholar), N V Ravi Kumar (Professor)



Extending the concept of “High Entropy Alloys (HEA)” to ceramic systems, a novel multicomponent oxide consisting of MgO, NiO, CuO, ZnO and CoO was synthesized into a single-phase rock salt structure named “Entropy Stabilized Oxides (ESO)”1. The presence of four or more cations allows properties to be tailored, that are otherwise difficult to obtain in conventional binary oxide systems. Several distinctly unique properties have been reported for this material composition and one such is high ratio of elastic modulus to thermal conductivity (E/?). The ratio of E/? for ESO is comparable or surpasses some of the prominent existing thermal barrier coating (TBC) materials (E/? ratios for BaZrO3, ESO and La2Zr2O7 are 40, 60 and 90 GPa W-1mK respectively)2 suggesting the potential of ESOs for TBC applications. However, the high temperature phase transformation and stability studies for these compositions have not been investigated or rather limited. In this work, we report a low temperature solution combustion synthesis (SCS) to produce nanocrystalline ESO. Systematic heat-treatment studies on powder samples were done in the temperature range of 500 – 1000°C in atmospheric ambience. X-ray diffraction and TGA studies were done to understand the influence of local stress field on the phase evolution & stability, complemented with molecular dynamics simulations. The nanocrystalline powder particles were subsequently sintered to near-theoretical density using spark plasma sintering (SPS). A comprehensive characterization on the effect of phase segregation on the thermal properties and mechanical stability of the sintered samples yield interesting propositions towards materials design.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 2169

# A multi-component ceramic coating for high-temperature application of light thermal protection material


Presenting author: Prof TONG-QI Li
Affiliation: Science and Technology on Advanced Functional Composites Laboratory, Aerospace Research Institute of Materials & Processing Technology
No.1, Nandahongmen Road,Fengtai District,100076 Beijing (Beijing, China)

Co-authors: YU-FENG Liu (Science and Technology on Advanced Functional Composites Laboratory Aerospace Research Institute of Materials & Processing Technology), ZHONG-WEI Zhang (Science and Technology on Advanced Functional Composites Laboratory Aerospace Research Institute of Materials & Processing Technology), ZHENG-HUI Xu (Science and Technology on Advanced Functional Composites Laboratory Aerospace Research Institute of Materials & Processing Technology), DA-HAI Zhang (Science and Technology on Advanced Functional Composites Laboratory Aerospace Research Institute of Materials & Processing Technology), ZHI-HAI Feng (Science and Technology on Advanced Functional Composites Laboratory Aerospace Research Institute of Materials & Processing Technology)



Carbon bonded carbon fiber (CBCF) material is a light thermal protection material (TPM) developed recently and can be used at higher temperatures compared to ceramic titles, which usually cannot be used above 1500? due to the degradation of their inner ceramic fibers. Oxidation protection of the CBCF is a key point for their application in oxidative atmospheres, now that carbon materials tend to be oxidized or ablated and then their mechanical performance will be decreased rapidly. In this work, a rapid coating-sintering method was adopted to prepare a multi-component ceramic coating consisting of HfB2, MoSi2 and SiC (HMS) for a CBCF. Electric arc tunnel (ET) was used to value the oxidation protection performance under the heating flux of 2.2MW/m2. It has been found that the multi-component ceramic coating has good compatibility with the CBCF and compact surface without micro-cracks. During ET testing, the coating behaved excellent, although micro-cracks appeared. The formed SiO2 filled in the micro-cracks and protected the CBCF from oxidation. The maximum temperature of the font surface of the test sample is about 1600? and that of its back surface is about 800?. Comparatively, the sample protected with other coating containing cracks behaved continuous temperature increasing of its back surface up to 1200? after 300s, although the front surface temperature was almost the same to the HMS coated sample. The oxidation of the carbon occurred during testing and reaction heat increased the back temperature. The weight reduction of the sample protected with the cracked coating also validates the oxidation reaction in it. As a conclusion, the HMS coating can protect the CBCF from oxidizing or ablating and the rapid

coating-sintering method is effective to prepare multi-component ceramic coating for high-temperature application of CBCF in oxidative atmospheres.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 2606

# Investigation and Process Development of Electroplated NiCo-Al2O3 Alloy Composites


Presenting author: Dr SANPO Noppakun
Affiliation: SCG Chemicals Co., Ltd
271 Sukhumvit Road,Map Ta Phut, Muang District,21150 Rayong (Thailand)
Co-authors: KOICHI Fukuda (SCG Chemicals Co. Ltd)



Bath electroplating of NiCo and NiCo-Al2O3 have been examined comprehensively and systematically with respect to different process parameters. Current density is found to be important parameter that control the content of the particles in the deposit and the mechanical property of the coating. The current bath electroplating protocol leads to uniform coating and well-dispersed Al2O3 nano-particle (agglomerates). The optimum current density and solid loading are 30 g/l and 2 A/dm2, although lower solid loadings do not alter the results significantly.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 2027

# Fabrication of Yb2Si2O7/SiC Composites for Self Healing EBC material


Presenting author: Prof NAKAYAMA Tadachika
Affiliation: 1603-1 Kamitomioka
Nagaoka Univ of Tech,940-2188 nagaoka (Niigata, Japan)

Co-authors:



In this research, Yb2Si2O7-Yb2SiO5-SiC composite, a candidate for top coat materials of environmental barrier coating (EBC) for gas turbine engine blades, was synthesized. This composite was confirmed that having an excellent self-crack-healing ability, due to the combination of various morphology (whisker, particulate) of SiC nanofillers, and the volume expansion of Yb2Si2O7 when they are transformed from Yb2SiO5 at high temperature. However, once all the healing agents in the composite have reacted, the self-healing ability cannot be continued. In a real gas turbine system, the turbine blades are required to operate through many cycles and thousands hour. If the composite cannot regenerate the healing agent continuously, the self-healing property would be impractical. In order to retain this ability, in this research, we tried to regenerate Yb2SiO5 through a thermal process in water vapor. The experimental results showed that healing agent can be regenerated partly after the heat treatment. This opens a new door for the development of self-healing composite materials.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 1742

# Thermodynamic calculation of CVD Yttrium Silicate from Y2O3-CH3SiCl3-CO2-H2-Ar System


Presenting author: Dr HE Fang
Affiliation: Northwestern Polytechnical University
127 West Youyi Road,Beilin District,Xi An, SN 29 Xi'an (Shaanxi, China)

Co-authors: LIU Yongsheng (Northwestern Polytechnical University), GAO Yuqing (Northwestern Polytechnical University), CHENG Laifei (Northwestern Polytechnical University)



SiCf/SiC composites have many excellent properties, such as low density, high strength and oxidation et al, which led to the widely application in aero-engine. However, the reaction between SiC and water limited the service life of this materials. We propose the idea of preparing yttrium silicate-modified SiC/SiC composite matrix by CVD from Y2O3-CH3SiCl3-CO2-H2-Ar system at a lower temperature. The effect of deposition temperature, pressure, reactant ratio were investigated. The results indicated that the most important factor is the proportion of CO2 in the gas source. As the proportion of CO2 increases, the amount of yttrium silicate increases. When n(CO2)/n(Y2O3+CH3SiCl3)?3, the yttrium silicate product yield tends to be stable. Under the condition of ensuring sufficient CO2, the H2 dilution ratio and the total system pressure have little effect on the amount of yttrium silicate, but in order to reduce the by-products production, we choose to control the total system pressure between 0.1~0.3 atm and the dilution ratio between 8~10. The temperature change causes a change in the crystal form of strontium silicate. And the amount of CO2 is sufficient, the temperature is higher than 1000oC, the formation of by-products can be reduced. Obviously, the value of n(Y2O3)/n(Y2O3+CH3SiCl3) has a great influence on the amount of yttrium silicate. When the temperature is higher than 1000oC, the yield of yttrium silicate increases with the increase of n(Y2O3)/n(Y2O3+CH3SiCl3). However, after the value of n(Y2O3)/n(Y2O3+CH3SiCl3) is increased to 0.4, due to the limitation of the software database, we cannot determine the crystal form and amount of yttrium silicate at this time. This requires experimental judgment. Subsequently, according to the thermodynamic calculation results, X1-Y2SiO5 and ?-Y2Si2O7 were successfully deposited at a lower deposition temperature of 1100oC, a system pressure of 2000Pa, a flow of CH3SiCl3 of 39ml/min, a H2 dilution ratio of 9, and a ratio of n(CO2)/n(H2) of 3:10.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 2310

# Processing of dense rare-earth silicates used as environmental barrier coating


Presenting author: M MOURET Thibault
Affiliation: Laboratoire des Composites ThermoStructuraux
3 allée de la Boétie,33600 Pessac (France)

Co-authors: REBILLAT Francis (Laboratoire des Composites ThermoStructuraux), MAILLE Laurence (Laboratoire des Composites ThermoStructuraux)



To be used in hot-section component of gas turbines engines, ceramic matrix composites (CMCs) as SiC/SiC have to be covered by an environmental barrier coating (EBC) to avoid oxidation and volatilisation of silica under moist environment. The relative thermomechanical and thermochemical compatibility of rare earth silicates with respect to silicon carbide, are responsible nowadays, of the trend to use them as EBC. Indeed, studies have shown the interest of rare earth disilicates as material to strongly limit the oxygen and water vapour diffusion until the composite.

This work aims to identify new solutions to improve the performances of rare earth disilicates as anti-diffusion layer, by reducing the residual porosity and the permeability of the material. As they are refractories material (Tm > 1700°C), the densification of rare earth silicate requires a high temperature, which promotes grain growth and consequently, oxygen and water vapour diffusion. Sinterings at low temperature (T < 1400°C) without applied pressure are carried out with the help of thermodynamics equilibriums providing a liquid phase, to get dense rare earth silicates. Literature shows that the addition of a third element as alumina in the RE2O3-SiO2 system (RE = Rare Earth), allows to strongly decrease the melting point of the component (RE = Y ? ? 1400°C). Thermodynamic calculations, reaction mechanisms and different compositions in the ternary RE2O3-SiO2-Al2O3 will be considered to obtain a liquid phase to improve the densification of the EBC.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 2130

# Development of Environmental Barrier Coatings on Ceramic Matric Composites assisted by LASAT (Laser Shock Adhesion Test)


Presenting author: Dr GUIPONT Vincent
Affiliation: MINES ParisTech - PSL / Centre des Matériaux
63-65 Rue Desbruères,91000 Evry (France)

Co-authors: COTTIN Guillaume (MINES ParisTech - PSL / Centre des Matériaux), MAUREL Vincent (MINES ParisTech - PSL / Centre des Matériaux), BAKAN Emine (2Forschungszentrum Jülich GmbH Institute for Energy and Climate Research (IEK-1), VAßEN Robert (Forschungszentrum Jülich GmbH Institute for Energy and Climate Research (IEK-1), KOCH Dietmar (3German Aerospace Center (DLR), Institute of Structures and Design)



Silicon carbide (SiC) fiber reinforced SiC ceramic matrix composites (CMCs) are envisaged as structural material for hightemperature parts in next generation aero-engines. A lower density and a higher thermal stability make such composite very attractive for industrial applications but there is a need for a protective coating to prevent the chemical degradation under service conditions. Therefore, Environmental Barrier Coating (EBC) systems consisting of a silicon bond coat and a rare-earth silicate top coat layer deposited by thermal spraying are envisaged and already successfully tested in gas turbine engines. In this work, two different Si/Yb2Si2O7 EBC samples were prepared by plasma spraying involving APS and High-Velocity APS respectively, each exhibiting a different deposition temperature. The non-contacting LASAT method (Laser Shock Adhesion Test) was implemented in order to estimate the potential of this laser shock method and its capability to support the development of optimized spraying parameters leading to enhanced interfacial properties. The LASAT method was previously developed on metal/ceramic systems for thermal barrier coatings (EB-PVD, APS) and also for hydroxyapatite coatings for biomedical applications. This first attempt on SiC/SiC composite substrate coated with an EBC by plasma spray was successful and revealed that the weakest region was located in the silicon layer near the ceramic top-coat. Further observation of damaged regions after laser shock confirmed that the debonding threshold that corresponding to the minimum of laser energy needed to create an interfacial crack was much higher for the APS coating deposited at a higher temperature. Further work is on progress to confirm the role of the deposition temperature on the resulting interface strength and also to address the influence of the composite thickness on the shockwave propagation.

Topic: 8 - Advanced Thermal and Environmental Barrier Coatings: Processing, Properties, and Applications
Abstract no. 2030

# Development of hafnia-based materials as potential environmental and thermal barrier for thruster application

Presenting author: Mme SéVIN Louise
Affiliation: ONERA/DMAS – University of Paris-Saclay
29 avenue de la Division Leclerc,92322 Chatillon (France)

Co-authors: JUSTIN Jean-François (ONERA/DMAS – University of Paris-Saclay), RAZAFINDRAMANANA Volatiana (Propulsion Office CNES Toulouse 31410 France.), MAUVY Fabrice (ICMCB Université de Bordeaux Pessac 33600 France), JULIAN-JANKOWIAK Aurélie (ONERA/DMAS – University of Paris-Saclay F-92322 Châtillon France.)

Anhydrous hydrazine, widely used as monopropellant in spacecraft thrusters, has been classified as toxic, mutagen and carcinogen and could be prohibited by the European Regulation REACh in the years 2020s. There by, CNES has engaged the development of a new low-toxicity and high performance green propellant which induces the study of new combustion chamber materials due to harsh operating conditions (flame temperature of 3000 K with oxidising combustion gases). Indeed, current engine materials were developed to withstand levels around 1700 K and solutions to reach 2500 K were all unsuccessful.
One part of the designing of such a material is focused on the development of a 3000 K resistant ceramic oxide as environmental and thermal barrier. Thanks to its high melting point, low thermal conductivity and relatively low thermal expansion, the cubic-stabilized HfO2 seems to be one of the most promising candidates. The cubic phase stability, obtained through doping with rare earth oxide3, is essential to avoid phase transformation and thus, minimizing cracks formation during thermal cycling. However, doping generates oxygen vacancies leading to physicochemical properties variations. Thus, the influence of various natures (Dy2O3, Er2O3, Gd2O3, Lu2O3, Y2O3 and Yb2O3), rates (9-33 mol. %), and associations of rare earth oxides was studied on thermal expansion (375-1873 K), on ionic conductivity (600-1150 K) and on thermal conductivity. The ionic conductivity decreases from 1.3.10-2 to 4.0.10-3 S/cm at 1150 K when the Y2O3 rate increases from 12 to 20 mol.%, whereas thermal expansion increases. Moreover, the lower is the ionic radius, the higher the thermal expansion. Considering inherent properties of Rare Earth as ionic radius, binding energy, molecular weight, ternary oxide systems will be investigated to fulfil most of the application requirements, promoting low thermal expansion and low ionic and thermal conductors material.
In the next months, properties as toughness and Young's modulus will be assessed to confirm the improvement brought by the co-doping solution.

# Topic: 9

# Polymer Derived Ceramics and Composites (incl. Reinforced foams)

Topic: 9 - Polymer Derived Ceramics and Composites (incl. Reinforced foams)
Abstract no. 2101

# Polymer-Derived Ceramic Nanocomposites for Applications at High Temperatures and in Harsh Environments

Presenting author: Dr IONESCU Emanuel
Affiliation: Technische Universität Darmstadt, Fachbereich Material- und Geowissenschaften
Otto-Berndt-Str. 3,64287 Darmstadt (Germany)

Co-authors:

Polymer-derived ceramic nanocomposites (PDC-NCs) can be synthesized via thermal conversion of suitable single-source precursors, leading in a first step to amorphous single-phase ceramics, which subsequently undergo phase separation processes to furnish bi- or multiphase ceramic nanocomposites. PDC-NCs have been shown to be excellent candidate materials suitable for applications at (ultra)high-temperatures and under harsh environments. In the present work, amorphous SiMC-, SiMCN- and SiMBCN-based materials (M = Ti, Hf, Ta) were synthesized via cross-linking and ceramization of tailor-made single-source precursors. High-temperature annealing of the obtained amorphous ceramics led to PDC-NCs with promising compositions, such as SiC/MC, MN/Si3N4/SiBCN or MC/MB2/SiC. The presented results emphasize a convenient preparative approach to nano-structured (ultra)high-temperature stable materials starting from greatly compliant single-source precursors. Recent results concerning the stability of the prepared ceramic nanocomposites in ultraharsh environments (i.e., oxidative atmosphere, combustion atmosphere, hydrothermal environment) will be highlighted and discussed.

Topic: 9 - Polymer Derived Ceramics and Composites (incl. Reinforced foams)
Abstract no. 1957

# Process technologies and applications of Basalt fiber reinforced SiOC composites


Presenting author: Prof GADOW Rainer

Affiliation: Institute for Manufacturing Technologies of Ceramic Components and Composites (IMTCCC) - University of Stuttgart
Allmandring 7b,70569 Stuttgart (Germany)

Co-authors: WEICHAND Patrick (Institute for Manufacturing Technologies of Ceramic Components and Composites (IMTCCC), - University of Stuttgart)



Polymer Matrix Composites (PMC) are widely used in lightweight engineering applications. The manufacturing technologies are fully developed and raw materials are cheap. Excellent mechanical properties in combination with low density qualify them as an ideal lightweight material. The limiting factors of these reinforced polymers are the useable service temperatures well below 250 °C and poor tribological properties. Promising lightweight composite materials, bridging the gap between PMC and CMC, are manufactured as polymer derived ceramics by the use of polysiloxanes and basalt fibers. Such competitive free formable Hybrid Composites are capable for lightweight applications in a temperature range between 300 and 850 °C and short time exposure up to over 1000 °C, even in oxidative atmosphere.
In order to qualify the material for series applications, manufacturing technologies like Resin Transfer Moulding (RTM), filament winding, Pultrusion or pressing techniques are employed. Cheap raw materials in combination with performing manufacturing technologies can establish completely new markets for these intermediate temperature composites. The special densification effect in conical extrusion nozzles under sumultaneous curing and heating of pulltruded profiles leads to dense composite structures with high fiber volume content. The pultruded products feature high strength and toughness. Sub micron fine filler powders have been introduced to further reduce the shrinkage of the matrix as well as subsequent re-impregnation by low vicosity resins.
Beside an increased thermal stability compared to polymeric composites, the Hybrid Composites show excellent tribological properties. An adjustable value of hardness and coefficient of friction open up a wide variety of friction applications fromultra light weight structures for bikes and electrically driven cars to heavy industrial equipment as brake linings for modern skyscraper lift systems. The presentation will comprise a detailed view on the manufacturing processes, a comprehensive (raw-) material characterization and will discuss already proven industrial applications.

Topic: 9 - Polymer Derived Ceramics and Composites (incl. Reinforced foams)
Abstract no. 2172

# New polysiloxane/polysilazane precursors for Digital Light Processing 3D Printing of Functional Ceramics

Presenting author: Prof KONG Jie
Affiliation: Northwestern Polytechnical University
Youyi West Road No. 127,710072 Xi'an (China)
Co-authors: ZHOU Rui (Northwestern Polytechnical University)

We developed a low-cost and simple synthesis method for UV curable polysiloxane and polysilazane preceramic precursors. Complex geometric polymer structures can be obtained by using Digital Light Processing (DLP) 3D printing method at high speed. After pyrolysis, the precursor-derived ceramic parts with linear shrinkage and high density were produced without any crack. An excellent microwave absorption performance were successfully achieved. The ceramic parts pyrolyzed from polysiloxane precursors show splendid thermal stability in air (T?900 ?, mass change < 2%; T?1400 ?, mass change < 5%). These preceramic precursors possess great potential 3D ceramic components with precision structure and desirable properties.

Topic: 9 - Polymer Derived Ceramics and Composites (incl. Reinforced foams)
Abstract no. 2165

# Role of SiC Fibers for Next Generation Applications


Presenting author: Dr HUMBS Werner
Affiliation: BJS Ceramics GmbH
Siemensstrasse 6a,86368 Gersthofen (Germany)
Co-authors: ROTHMANN Michael (BJS Ceramics GmbH)



Ceramic Matrix Composites (CMC) are a key technology to fulfill future reduced emission requirements in next generation applications such as aero engines. The CMCs consist of a SiC fiber re-inforced SiC matrix, resulting in a so-called SiC/SiC composite. The role of a SiC fiber is many-faceted, with unique mechanical and thermal properties, but availability and export control considerations are equally important. Current production of commercial SiC fibers is centered in Japan and in the USA, however all commercial SiC fibers currently use the same raw material source. Since the material class is an export controlled good, a regional supply chain is an important element. For more than 12 years, SiC fibers have been developed in Germany using locally sourced raw materials. This development is now in its final stage under the direction of BJS Ceramics GmbH. An update on the status of our own SILAFIL® SiC fiber development will be presented. The influence of various processing parameters on the fiber properties will be discussed. We will also give an overview of the competitive properties of our SiC fibers. Finally, we will present an outlook on the transfer of our lab-scale technology into pilot-scale, which will start from mid 2019.

Topic: 9 - Polymer Derived Ceramics and Composites (incl. Reinforced foams)
Abstract no. 1930

# Innovative synthesis and characterization of large h-BN single crystals: from bulk to nanosheets


Presenting author: Dr TOURY Bérangère
Affiliation: University of Lyon
06 rue Victor Grignard,69100 Villeurbanne (France)

Co-authors: LI Yangdi (INSA Lyon), JOURNET Catherine (University of Lyon), GARNIER Vincent (INSA Lyon), STEYER Philippe (INSA Lyon)



Prompted by the rising star of graphene, 2D nanomaterials are now a hot issue in the scientific world. Among them, the h-BN nanosheet (BNNS), consisting of thin atomic layers made of B and N atoms covalently bounded, is particularly relevant. Actually, BNNS has shown to be an excellent gate dielectric support for graphene and other two-dimensional materials owing to its atomically smooth surface, high thermal conductivity and stability combined with high mechanical strength. Compared with conventional SiO2 substrate, lattice matching and absence of dangling bonds make BNNS and graphene excellent pairing materials and give incentive to develop various Van der Waals hetero-structures. However, it has to be pointed out that such applications cannot be put into use without high purity large BNNSs.
In order to achieve high quality and large BNNSs, we propose two novel synthesis ways by the Polymer Derived Ceramics (PDCs) route involving polyborazylene as precursor, combined with different sintering techniques. These promising approaches allow synthesizing pure and well-crystallized h-BN single crystals, which can be easily exfoliated into BNNSs with lateral size over hundreds microns. Here we present recent investigations on how to optimize processes, considering the influences of both sintering temperature (1200°C to 1950°C) and crystallization promoter ratio (0 to 10wt%) on h-BN. Structural studies were led by TEM and Raman spectroscopy. Both methods evidence a very high crystalline quality attested by the LWHM value, 7cm-1, as the best reported in literature. More original characterizations were carried out by cathodoluminescence and XPS to prove the high BNNSs purity from both structural and chemical point of view. As a final application purpose, further physical measurements have confirmed that derived BNNSs exhibit an interesting dielectric constant of 3.9 associated with a dielectric strength of 0.53 V/nm.

Topic: 9 - Polymer Derived Ceramics and Composites (incl. Reinforced foams)
Abstract no. 1917

# Thermodynamic Modelling Applied to Ceramic Systems


Presenting author: Prof KUMAR K.C. Hari
Affiliation: Metallurgical & Materials Engineering, Indian Institute of Technology Madras
Sardar Patel Road,Adyar,600036 Chennai (Tamil Nadu, India)

Co-authors:



It is widely recognised that the phase diagrams and thermochemical data are essential to answer many practical questions that arise in the development of materials and their processing. The CALPHAD method, a computer-assisted calculation method, which offers a cost-effective and efficient alternative to conventional methods to obtain much-needed phase diagram and thermochemical information in multicomponent systems. Heart of the Calphad method is the Gibbs energy models for phases. Statistical estimation of the required Gibbs energy model parameters is accomplished through a combinatorial approach involving carefully selected experimental data and results from theoretical estimation techniques. Main outcome of such an exercise is a set of internally consistent Gibbs energy functions for various phases that describes the material system. Powerful computer programs in combination with reliable Gibbs energy functions stored in the databases, allow calculation of phase diagrams and thermochemical properties that suites the need. In this talk I will discuss the application of the Calphad method of thermodynamic modelling applied to ceramic systems, mainly drawing examples from our work.

Topic: 9 - Polymer Derived Ceramics and Composites (incl. Reinforced foams)
Abstract no. 1895

# Organomorphic Composites as a New CMC Type for Aerospace and Aircraft Application


Presenting author: Dr BOGATCHEV Evgheniy
Affiliation: JSC Kompozit
4, Pioneerskay,141070 Korolev (Moscow region, Russia)

Co-authors:



Increase of CMC microstructure uniformity is a novel trend and a very important for the aerospace and aircraft application development. The organomorphic composites (OC) have properties which are traditionally difficult for CMC: gas tightness (Darcy coefficient on the level 10-18-10-19 m2) and very low roughness of polished surface (up to Ra 0,1). These properties are provided by initial network which is formed of appressed during pyrolysis polyacrylonitrile, polycarbosilane or polysilazane cured filaments, therefore the small and relatively uniform gaps between them (as rule from few micrometers to several tens micrometers) are specific for the network. The coherency of the dense (sometimes up to 0.4 of the density of ceramics fibers) pyrolyzed network is achieved by a diffusive interaction of the filaments at the places of their contacts as far back as the polymer state. This mechanism is discussed in the talk on the base of scanning electron microscopy, mass-spectroscopy, thermogravimetric and elementary analysis data.
Network densification has been carried out with SiC by PIP and CVI methods. The properties of ceramics fibers produced in situ within the network provide well mechanical OC characteristics and high temperature long-term oxidation resistance at 1400oC (especially for OC on the base of SiC- ? SiCN-fibers). Integration of the ceramics fibers and reinforcing network manufacturing within the single technologic stage essentially decreases the cost of organomorphic CMC and articles of them.

Topic: 9 - Polymer Derived Ceramics and Composites (incl. Reinforced foams)
Abstract no. 2288

# XCT Investigation of Void Formation During First Pyrolysis of a PIP Processed CMC

Presenting author: Dr KING Derek
Affiliation: UES, Inc.
4401 Dayton-Xenia Rd,45432 Dayton (OH, United States)

Co-authors: KEY Thomas (UES Inc.), PRZYBYLA Craig (Air Force Research Laboratory), CINIBULK Michael (Air Force Research Laboratory)

Ceramic matrix composites (CMCs) that are processed using the polymer infiltration and pyrolysis (PIP) method experience mass loss and volume shrinkage of the polymer based matrix during the pyrolysis conversion to a ceramic. In this study, a model CMC was engineered with a fiber, inactive matrix filler, and pre-ceramic polymer enabling X-ray computed tomography (XCT) analysis, with each phase selected for characteristically different X-ray transmission behavior. Tomography scans of the same area for a specific CMC were performed after autoclave consolidation and after pyrolysis to temperatures of 800, 1200, and 1600°C. The resulting scans provided insight into the three-dimensional structure of voids in the CMC, and the evolution of the void network during the initial pyrolysis. Delaminations between fiber plys became apparent as pyrolysis temperature was increased, leading an increased void network connectivity. Phase distribution, evolution of the void network, and the effect of fiber tow placement on the observed void network will be discussed.

Topic: 9 - Polymer Derived Ceramics and Composites (incl. Reinforced foams)
Abstract no. 2294

# Cyclic thermal aging of alumina fiber reinforced SiCO composites for applications in power plants


Presenting author: Mme WOLFF Nadja
Affiliation: Universität Bayreuth
Ludwig-Thoma-Straße 36 b,95447 Bayreuth (Germany)

Co-authors: LANGHOF Nico (Universität Bayreuth), KRENKEL Walter (Universität Bayreuth)



In modern power plants higher pressures > 250 bar and temperatures above 550 °C are requested for hot steam pipes in front of the steam turbine. Due to this demand, a higher creep stability is required, which conventional low alloyed steels and chromium alloyed steels cannot provide. Fiber reinforced Oxide Ceramic Matrix Composite (OCMC) jackets for pressure vessels are a promising efficient alternative for life time extension and temperature increase.
The possible on-site fabrication by filament winding and low pyrolysis temperatures needs a special material composition that means pure alumina fibers reinforcement in a polymer derived SiCO matrix, pyrolysed up to 750 °C.
Within this study, the matrix is derived from two polysiloxanes. Because of the application related, but low pyrolysis temperature of just 750 °C the long term thermomechanical and oxidation stability of the SiCO-matrix are in the focus of this work.
Additionally a continously changing temperature curve with high heating rates was designed, in order to simulate the conditions in power plants. Thus, this special cyclic heating profile is denoted by temperatures up to 550 °C in air. Changes in porosity, young's modulus, tensile strength and shear strength by this thermal aging were studied. During the first 24 hours the porosity and tensile strength increase by oxidation and the shear strength is reduced. At longer aging times the tensile strength, strain to failure and porosity decreases slightly because of re-densification. The effect of cyclic heating and cooling on the young‘s modulus was studied by the impulse excitation method.
New aspects of this work are the low pyrolysis temperature of 750 °C, that is leading to a not oxidation stable incorporation of carbon. This leads to a metastable behavior of the matrix and to application relevant transformations in the composite during aging.

# Mechanical characterization of spark plasma sintered precursor derived transition metal oxide (TMO)-silicon oxycarbide nanocomposites

Presenting author: M ERANEZHUTH Wasan Awin
Affiliation: Research scholar
Department of MME,IIT Madras,600036 Adayar (Tamil Nadu, India)
Co-authors: BERNARD Samuel (Scientist), N.V Ravi Kumar (Professor)

In recent years, silicon oxycarbide (SiOC) based ceramics have gained a lot of attention in the research community due to their excellent mechanical properties as well as their high chemical and thermal stability.[1] SiOC based ceramics have been synthesized through a wide variety of processing routes, out of which commonly used techniques are the sol-gel[2] and the Polymer-Derived Ceramic route (PDC).[3] The main advantage of implementing PDC route is in the flexibility to control and adapt the structure and the composition of the ceramics for the targeted application based on the chemistry of the selected preceramic polymer as precursor.[4] The development of nanocomposites in which the structure of at least one phase is designed at nanoscale allows performances that can reach far beyond those of conventional PDCs.[5]–[8] As an illustration, the design of transition metal oxide (TMO) based SiOC ceramics, can have a profound influence in the field of catalysis, dielectrics, batteries and supercapacitors due to the combinatorial effect exhibited by SiOC and TMO phases. Understanding the mechanical stability of these materials is of profound importance to ensure long term reliability and practical viability.

In this work, the mechanical properties of in situ crystallized TMO-SiOC nanocomposites (TM = Ti, Zr, Hf, and Nb) synthesized via polymer derived ceramic route (PDC) were investigated. Dense monoliths were prepared using spark plasma sintering (SPS) and the apparent density was measured using water displacement method. The phase evolution was understood and crystallite size quantified using X-ray diffraction (XRD). The hardness and elastic modulus were determined using nanoindentation and the fracture toughness was calculated using indentation crack length method. In addition to this, ball-on-three ball (B3B) technique was used to evaluate the biaxial flexural strength and fractographic studies were carried out to understand the fracture mechanisms.

Topic: 9 - Polymer Derived Ceramics and Composites (incl. Reinforced foams)
Abstract no. 1826

# Synthesis of Poltcarbosilane by new methotorogy with possible uses for matrix source of CMC


Presenting author: Dr NARISAWA Masaki

Affiliation: Graduate School of Engineering, Osaka Prefecture University
1-1, Gakuen-Cho,599-8531 Sakai (Osaka, Japan)

Co-authors: KOUYA Yamada SAKURA Ukyo INOUE Hirofum



Polycarbosilane (PCS) is known to be derived from polydimethylsilane (PDMS) by thermal condensation reaction in an inert atmosphere at temperatures of 400-450 ºC in general. In this process. Si-Si bonds in PDMS are converted to Si-C bonds by methyl group insertion. Yield of thus obtained PCS is, however, usually at the range of 50-60 mass%. Recently, we began to investigate the effect of CO2 coexistence on the condensation reaction of PDMS. Si-C bond formation process was accelerated at 350-360 ºC, and brown viscous liquid "PCS" was obtained at 380 ºC. The yield of thus obtained "PCS" depended on the applied CO2 pressure strongly. The residual polymer yield increased from the original 60% (in an autoclave with a N2 atmosphere) up to >95% at the CO2 pressure of 10 bar. Apparent viscosity also increased by increasing the CO2 pressure. Spectrum analysis indicated the existence of Si-CH2-Si, Si-O-Si and a small amount of Si-H in the molecular structure. The local structure of the obtained polymer is similar to that of classic PCS, while liquid nature indicates the difference in long range ordering in the synthesized polymers. Although the ceramic yield of the viscous liquid polymer (heat-treated at 380 ºC) is limited at 30 mass%, the obtained polymers are promising as matrix source for CMC after suitable combination of additives and cross-linking agents. The obtained polymer yield, close to 100%, is highly attractive has for wide variety of industrial uses.

Topic: 9 - Polymer Derived Ceramics and Composites (incl. Reinforced foams)
Abstract no. 1887

# Porous catalytic active SiOC monoliths by in-situ formation of Ni nanoparticles in solution based freeze casting


Presenting author: M SCHUMACHER Daniel
Affiliation: Advanced Ceramics, University of Bremen, Germany
Am Biologischen Garten 2,28359 Bremen (Bremen, Germany)

Co-authors: WILHELM Michaela (Advanced Ceramics University of Bremen Germany), REZWAN Kurosch (Advanced Ceramics University of Bremen Germany)



Due to their unique properties, polymer derived ceramics (PDCs) have been studied for various applications. In-situ formation of metal nanoparticles enables the relatively new potential application of heterogeneous catalysis. Often, catalytic active PDCs were prepared in reflux synthesis resulting in microporous particles. Additional macroporosity would be beneficial to overcome mass transport limitation and to ensure a better accessibility of in-situ formed metal nanoparticles. This work reports the in-situ formation of Ni nanoparticles in solution based freeze casting in order to obtain catalytic active porous SiOC monoliths. Due to limitations in composition caused by the solution based freeze casting process, different cross-linking and complexing agents were carefully screened under the prerequisite of complexing ability and compatibility with the process. Prismatic pore structure was obtained for all samples and (3-Aminopropyl)triethoxysilane was found to be most efficient in creating well distributed Ni nanoparticles with an average size of 4 nm. Increasing ratio of complexing agent to Ni ions results in improved complexing of Ni ions with smaller Ni nanoparticles. Furthermore, the nickel source was changed from nickel acetylacetonate to nickel nitrate with a slight decrease in BET surface area. All samples exhibit hydrophobic surface characteristic with increasing hydrophilicity at higher complexing ratios. Additionally, high BET surface areas of around 400 m2g-1 are found for all samples. Catalytic tests using CO2 methanation are in accordance with the trends observed in Ni particle size and confirm that (3-Aminopropyl)triethoxysilane with nickel acetylacetonate and an increased ratio of complexing is most active in CO2 methanation showing a conversion of 50 % and a selectivity of 74 % at 400 °C. The combination of solution based freeze casting with metal containing PDCs allows for macroporous catalytic active SiOC monoliths. In a next step optimization towards heterogeneous monolithic catalysts is possible.

Topic: 9 - Polymer Derived Ceramics and Composites (incl. Reinforced foams)
Abstract no. 2019

# Development of Functional Superparamagnetic SiCN Ceramic Fibers

Presenting author: Dr VIARD Antoine
Affiliation: University of Bayreuth
Ludwig-Thoma-Strasse 36B,95447 Bayreuth (Bavaria, Germany)

Co-authors: KURZ Hannah (University of Bayreuth), LOPEZ-FERBER David (University of Limoges), HEYMANN Lutz (University of Bayreuth), WEBER Birgit (University of Bayreuth), BERNARD Samuel (University of Limoges), KRENKEL Walter (University of Bayreuth), MOTZ Günter (University of Bayreuth)

Ceramic fibers are key structural components in Ceramic Matrix Composites (CMCs). Especially silicon-based non-oxide ceramic fibers exhibit enhanced mechanical and thermo-structural properties, which are crucial for applications under harsh conditions. Due to the highly sophisticated production process of these ceramic fibers, the incorporation of new functionalities is very challenging. However, it would allow for the development of the next generation CMCs exhibiting new features as defect detection or magnetic shielding.

The aim of the presented work was the development of SiCN ceramic fibers with superparamagnetic properties. Based on the fiber processing strategy we developed from commercial available organosilazanes Durazane 1800 and Durazane 1033 in the past, the polymer precursors have been chemically modified with an appropriate iron complex in order to obtain an iron-modified meltable polysilazane. Adjusted chemical engineering of both metal complex and polysilazane ensures adequate rheological properties of the iron-modified preceramic polymer for a stable melt-spinning process, thus enabling the processing of green fibers. After curing and pyrolysis of the polymer fibers, Fe-modified SiCN ceramic fibers with good mechanical properties were obtained. The homogeneously dispersed Fe-containing nanoparticles generated during pyrolysis confer superparamagnetic properties to the resulting fibers. Such functionalization strategy can be further extended to other elements, enabling the incorporation of different features.

Topic: 9 - Polymer Derived Ceramics and Composites (incl. Reinforced foams)
Abstract no. 2284

# Novel ceramic matrix composites with tungsten and molybdenum fiber reinforcement


Presenting author: Dr FRIEß Martin

Affiliation: German Aerospace Center (DLR), Institute of Structures and Design
Pfaffenwaldring 38-40,70569 Stuttgart (Germany)

Co-authors: MAINZER Bernd (German Aerospace Center (DLR), Institute of Structures and Design), LIN Chaorong (TU Darmstadt Fachgebiet Disperse Feststoffe), RIEDEL Ralf (TU Darmstadt Fachgebiet Disperse Feststoffe), RIESCH Johann (Max-Planck-Institut für Plasmaphysik), FEICHTMAYER Alexander (Max-Planck-Institut für Plasmaphysik), FUHR Maximilian (Max-Planck-Institut für Plasmaphysik), ALMANSTöTTER Jürgen (Osram GmbH), KOCH Dietmar (German Aerospace Center (DLR), Institute of Structures and Design)



Damage-tolerant ceramic matrix composites (CMC) are prone to high temperature applications under severe environmental conditions and usually utilize carbon or ceramic fibres (e.g. SiC) as reinforcements of ceramic matrices with inherent low elongation to break compared to common metals.
However, CMC reveal an elongation to break and stiffness similar to the ceramic matrices, and thus need a fibre coating in order to improve the elongation to break length and thus to achieve damage tolerance of the composite. In addition, such fibers often expose a low ductility during failure. As a consequence, design criteria for components of such CMC materials are limited by the low strain of failure.
In order to overcome this problem, we follow the idea of a reinforcement concept of a ceramic matrix reinforced by refractory metal fibres to reach pseudo ductile behaviour during failure. Tungsten (W) and molybdenum (Mo) fibers were chosen as reinforcement in SiCN CMC manufactured by polymer infiltration and pyrolysis process. These fibres are commercially available since they are widespread used in light bulbs, etc. , and possess an intrinsic higher elongation to break, compared to ceramic fibres, as well as high stiffness even at high temperatures.
W/SiCN and Mo/SiCN composites were manufactured via filament winding and resin transfer moulding of commercially available polysilazanes, pyrolysed and re-densified by multiple reinfiltration and pyrolysis steps. These composites were investigated with respect to microstructure, flexural and tensile strength. Single fibre strengths for W and Mo were investigated and compared to the strength of the composites. Tensile strengths of 206 and 156 MPa as well as bending strengths of 427 and 312 MPa were achieved for W/SiCN and Mo/SiCN composites, respectively. W fibre became brittle across the entire cross section, while the Mo fibre showed a superficial, brittle reaction zone but kept ductile on the inside.

Topic: 9 - Polymer Derived Ceramics and Composites (incl. Reinforced foams)
Abstract no. 2251

# Metal-Catalyst-Free Access to Multiwall Carbon Nanotubes/Silica Nanocomposites (MWCNT/SiO2) from a Single-Source Precursor


Presenting author: Dr MERA Gabriela
Affiliation: TU Darmstadt
Otto-Berndt-Strasse 3,64287 Darmstadt (Hesse, Germany)

Co-authors:



Carbon nanotubes (CNTs) as important one-dimensional nanomaterials with outstanding electrical, thermal and mechanical properties, have a great potential for various applications in electronics, energy, composites, drug delivery, nanoimaging etc. Since the mechanical and electronic properties of CNTs strongly depend on their chirality, diameter, defect structure, etc., such nanotubes have entered the spotlight of nano-science. Typically, CNTs are produced via vapor-liquid-solid (VLS) processes catalyzed by 3d valence transition metals (Fe, Co and Ni), noble metals such as Au, Ag and Cu, as well as main group metals such as Pb and In. Unfortunately, the catalysts remain in the CNTs as undesired impurities after synthesis. The catalyst poisoning, as reported in several publications, constitutes the major drawback of this route due to the ability of the metals to decompose hydrocarbons and to induce the post-rearrangements of the CNTs.

Herein we are reporting for the first time on a ‘green’, metal catalyst-free in situ generation of bamboo-like multi-wall carbon nanotubes (MWCNTs) within a mesoporous silica-rich matrix during the thermal decomposition of a carbon-rich poly(organosiloxane)s. Poly(organosiloxane)s are precursors for polymer-derived silicon oxycarbides (SiOxC4-x), promising materials for high temperature application due to their high stability in harsh environments. X-Ray amorphous SiOxC4-x have unique structural features such as reduced mass fractal dimension and nano?heterogeneity, being considered intrinsic nanocomposites. Chemical design of polymeric precursors facilitates the formation of non-mixed bond-structured SiOxC4-x ceramics at 900°C in Ar atmosphere. The influence of initial structure of the precursor, the low temperature of the phase separation as well as the role of the of silica particle shapes, sizes and pore sizes on the formation and microstructural particulars of MWCNTs will be discussed. Uniaxial hot-pressing at 1600°C yields mechanically stable monoliths with electrical conductivity higher than 2000 S/m. These results will be highlighted and discussed within the context of prospective energy-related applications.

Topic: 9 - Polymer Derived Ceramics and Composites (incl. Reinforced foams)
Abstract no. 2067

# Polymer-Derived Si-B-C-N Fibers: From the synthesis of polymers with tuned melt-spinnable properties to the characterization of fibers


Presenting author: Dr BERNARD Samuel
Affiliation: CNRS-Institute of Research on Ceramics (IRCER)
CEC-12 rue Atlantis,87068 Limoges (France)

Co-authors:



development of materials in which compositions, shapes and textures are tuned on demand. Traditional techniques are energy-ineffective and severely limit the shape and texture complexities of the part which can be manufactured. Furthermore, the ability to control the purity and structure of the product is restricted. These inherent difficulties can be overcome by the development of synthetic paths where molecular chemistry and chemistry of materials are combined rationally. The Polymer-Derived Ceramics (PDCs) route represents one of these synthetic path solutions. The chemistry (elemental composition, compositional homogeneity and atomic structure), the processing properties and the reactivity (thermal and chemical) of related polymers can efficiently be controlled and tailored to supply, after shaping and pyrolysis processes, non-oxide ceramics with the desired compositional phase distribution and homogeneity as well as shape. This concept has been applied to the preparation of simple carbides, nitrides of various main groups and transition elements as well as homogeneous mixtures or solid solutions of pseudo-binary combinations of ceramics and nanocomposites. Such a concept is particularly adapted to the preparation of Polymer-Derived Ceramic Fibers; in particular in the Si- and B-containing ternary and quaternary systems because of their covalent bonding and the poor mobility of boron and silicon in their respective nitride and carbide phases which provide high thermal and structural stability. This paper presents our last results on the design and characterization of Si-B-C-N fibers through the synthesis of preceramic polymers with tuned melt-spinnable properties.

Topic: 9 - Polymer Derived Ceramics and Composites (incl. Reinforced foams)
Abstract no. 1824

# Synthesis and Properties of Biologically-Inspired Pre-Ceramic Polymers


Presenting author: Dr DICKERSON Matthew
Affiliation: US Air Force
2941 Hobson Way,45433 WPAFB (OH, United States)

Co-authors: APOSTOLOV Zlatomir (US Air Force Research Laboratory), GUPTA Maneesh (US Air Force Research Lab), DENNIS Patrick (US Air Force Research Lab), SANFORD Maria (US Air Force Research Lab), CINIBULK Michael (US Air Force Research Lab), DICKERSON Matthew (US Air Force Research Lab)



The polychaete worms of the Nereididae family have distinct conical teeth that are lightweight and of a hardness comparable to the dentin of human teeth. Unlike most bone or teeth, polychaete teeth do not contain a mineral constituent. Instead, these biostructures consist of a histidine-rich protein cross-linked through metal coordination with zinc ions. This unique design of metal ion coordination with imidazole containing macromolecules to produce hard and lightweight materials offers a simple platform for synthetic polymer cross-linking. More specifically, imidazole or pyridine moieties can be chemically incorporated into preceramic polymer (polycarbosilane, polysiloxane) backbones or side-chains. Exposure of the functionalized preceramic polymer to a bath of metal ions allows for nearly instantaneous cross-linking to produce solid materials or fibers, contrasting from the time-consuming process of thermally curing these materials. In this work, the functionalization of polycarbosiloxanes with pyridine moieties is carried out through AIBN initiated thiol-ene click chemistry of 4-vinylpyridine with the thiol side chains in the polysiloxane polymers. The rheological behavior, ceramic yield, and material properties of these polymers and the effects of metal chelation will be discussed in this presentation.

Topic: 9 - Polymer Derived Ceramics and Composites (incl. Reinforced foams)
Abstract no. 2147

# Data Driven Material Models for Process Modeling of Preceramic Polymers


Presenting author: Dr KEY Thomas
Affiliation: U.S. Air Force Research Lab and UES Inc.
Area B, Builting 655,2230 Tenth St.,45433 WPAFB (Ohio, United States)

Co-authors: DEREK King (U.S. Air Force Research Lab and UES Inc.), DIPEN Patel (U.S. Air Force Research Lab and UES Inc.), WILKS Garth (U.S. Air Force Research Lab), APOSTOLOV Zlatomir (U.S. Air Force Research Lab), CINIBULK Michael (U.S. Air Force Research Lab)



Process modeling is necessary to improve manufacturing efficiency and reduce scrap rates of PIP based ceramic matrix composites. Hence, materials performance models are critical to capture the behavior of pre-ceramic polymers as a function of temperature and time. In particular, data driven models are developed and demonstrated for polycarbosilanes and polycarbosiloxanes that address evaporative mass losses and pyrolysis related changes in mass and density. This work will enable a robust prediction input parameters such as volumetric shrinkages needed for finite element models to simulate residual stresses in PIP based ceramic matrix composites.

Topic: 9 - Polymer Derived Ceramics and Composites (incl. Reinforced foams)
Abstract no. 2175

# Phase and nanostructure evolution in zirconium modified polymer derived silicon carbonitride ceramic hybrids

Presenting author: M ANAND Rahul
Affiliation: National Institute of Technology Rourkela, Odisha, India
Room No. - B115,H B Hall of Residence, NIT Rourkela,769008 Rourkela (Odisha, India)
Co-authors: BHUSAN NAYAK Bibhuti (Department of Ceramic Engineering National Institute of Technology Rourkela.), KUMAR BEHERA Shantanu (Department of Ceramic Engineering National Institute of Technology Rourkela.)

Si based polymer-derived carbide based ceramics, such as SiCN, SiBCN, are well known for their excellent thermal stability, creep and oxidation resistance. Their unique shaping advantages and easy modification of nanostructures make them suitable for barrier coatings at high temperature and/or harsh environments. Polysilazanes are unique Si based polymers that yield SiCN based amorphous ceramics, which possess better thermal stability as compared to SiOC ceramics. Incorporation of molecular sources of reactive elements, including Zr, Ti, or Hf, into the precursors alters the nanostructure of the hybrid ceramic, providing a unique advantage of tailoring the phase assemblage and oxidation resistance. The current work is based on the evolution of the nanostructure of the reactive element doped SiCN ceramics upon introduction of Zirconium as molecular source. An alkoxide of Zr incorporate the metal into commercially available polyvinylsilazane, and ceramized at temperatures ranging from 1000-1400 oC in flowing nitrogen atmosphere. The Zr incorporated SiCNO ceramic (SiZrCNO) remained essentially single phase amorphous at 1000 oC, However, with higher pyrolysis temperatures, phase separation of Zr into nanocrystals of tetragonal zirconia was observed and confirmed by XRD and TEM. The t-ZrO2 nanocrystals were exceptionally homogeneously distributed throughout the amorphous SiCNO matrix, as confirmed by high resolution TEM. The crystallite size of zirconia at different temperatures in the range of 1000-1400 oC was calculated using Scherrer formula, and found in the range of 2 to 9 nm. The pyrolyzed SiCNO ceramics exhibited amorphous nature even at 1400 oC, with prolonged heating schedule yielding precipitation of Si3N4 and SiC. The unique structure of ZrO2 nanocrystals embedded in the amorphous matrix of SiCN ceramics can be used as a bond coat between the carbide based substrates and oxide based top coats for high temperature applications. The retention of tetragonal ZrO2 in the ceramic matrix may help achieve enhanced toughness of the bond coat.

Topic: 9 - Polymer Derived Ceramics and Composites (incl. Reinforced foams)
Abstract no. 1825

# Synthesis of Multicomponent Polycarbosilane Systems for the Advanced Manufacturing of Polymer-Derived Ceramics


Presenting author: Dr DICKERSON Matthew

Affiliation: US Air Force
2941 Hobson Way,45433 WPAFB (Ohio, United States)

Co-authors: BALDWIN Luke (US Air Force Research Laboratory), RUESCHHOFF Lisa (US Air Force Research Laboratory), MARTIN Kara (US Air Force Research Laboratory), DENEAULT James (US Air Force Research Laboratory), CINIBULK Michael (US Air Force Research Laboratory), KOERNER Hilmar (US Air Force Research Laboratory), DALTON Matthew (US Air Force Research Laboratory), DICKERSON Matthew (US Air Force Research Laboratory)



Preceramic polymers (PCPs), including carbosilanes, are a unique group of materials that convert from their polymeric form to inorganic materials via heat treatment at high temperatures (>600 °C). PCPs are attractive for the fabrication of advanced ceramics, including SiC fibers and composite matrices, as they possess the ability to control final ceramic chemistry and nanostructure. The polymeric nature of PCPs enables the preparation of ceramic materials with geometries far more complex than those achievable by classical powder processing methodologies (e.g., hot pressing). In this presentation, we will present our recent efforts in the synthesis of a carbosilane-based PCP system that can be formulated to yield SiOC with further processing producing SiC. The PCP system investigated in this work consists of allyl terminated hyperbranched polycarbosilane polymers and a silane-rich small molecule crosslinking agent.  We will discuss how hyperbranched polycarbosilane can be successfully paired with reactive silane crosslinkers, cured, and thermally treated to generate polymer derived ceramics. Preliminary results on the printing of these precursors will be presented.

Topic: 9 - Polymer Derived Ceramics and Composites (incl. Reinforced foams)
Abstract no. 2105

# Polymer-derived SiC matrix composites for high temperature electrolysis: A Novel Catalyst for Electrochemical Water Splitting

Presenting author: Prof YU Zhaoju
Affiliation: College of Materials, Xiamen University, Xiamen 361005, China.
Si Ming Nan Lu,361005 Xiamen (China)

Co-authors:

Electrochemical water splitting is one of the most efficient ways to produce hydrogen due to its renewability and environmental-friendliness. Water splitting at low overpotentials to increase the efficiency of the process requires novel active catalysts for the hydrogen evolution reaction (HER). In this lecture, we report on the in-situ formation of a nano-sized Nowotny phase (NP) Mo4.8Si3C0.6 embedded in a porous SiC-based matrix via a novel single-source-precursor approach. The advanced features of our NP/C/SiC nanocomposite are as follows: i) in-situ formed catalytically active NP nanoparticles within an in-situ generated SiC/C-based matrix to avoid aggregation of the nanosized NP in the final ceramic matrix and ii) the simultaneous formation of carbon-rich phases improving the electric conductivity, which significantly increases the charge transfer rate during the HER process. As a result, the NP/C/SiC-based nanocomposites exhibit excellent HER activity with low overpotentials of 22 and 138 mV vs. reversible hydrogen electrode for driving cathodic current densities of 1 and 10 mA cm-2 in acidic media, respectively. The analyzed electrocatalytic performance exceeds that of most Mo2C-based electrocatalysts. Moreover, our electrocatalytic system shows outstandingly high stability ratio (over 90 %) during 35 h. To the best of our knowledge, this is the first time that a NP is discovered to possess electrocatalytic activity and excellent durability in terms of the HER. Our discovery extends and broadens the potential applications of polymer-derived ceramics in the field of energy materials.

Topic: 9 - Polymer Derived Ceramics and Composites (incl. Reinforced foams)
Abstract no. 2677

# Synthesis of Polycarbosilanes for characteristic applications


Presenting author: M MISHRA Raghwesh
Affiliation: DMSRDE, DRDO
DMSRDE,G T ROAD,208013 Kanpur (Uttar Pradesh, India)

Co-authors: GUPTA Rakesh K. (Defence Materials and Stores Research and Development Establishment Kanpur India), CHAKRABORTY Joy (Defence Materials and Stores Research and Development Establishment Kanpur India), KUMAR Suresh (Defence Materials and Stores Research and Development Establishment Kanpur India), RANJAN Ashok (Defence Materials and Stores Research and Development Establishment Kanpur India), PRASAD Eswara (Defence Materials and Stores Research and Development Establishment Kanpur India)



Polycarbosilane (PCS), an organosilicon polymer has been of great interest as precursor for silicon carbide for different high end applications such as structural material like SiC fibers, C/SiC and SiC/SiC composites in aerospace vehicles, SiC foams for thermal managements and precursor for high temperature SiC films and high temperature semiconductor devices. Though, the chemical nature of PCS remains similar for all the mentioned applications but its overall properties have to be different for different applications. Aim of the present study has been to synthesize different grades of PCS and explore their applications for development of SiC based products. Polydimethylsilane (PDMS) was synthesized by Wurtz condensation elimination reaction of dichlorodimethylsilane with molten sodium at 110?C for 8 h in presence of a suitable concentration of phase transfer catalyst. PDMS was converted into PCS by thermal backbone rearrangement process at normal atmospheric pressure. PDMS was heated slowly up to 450?C under inert atmosphere and at atmospheric pressure. Rate of heating, ultimate temperature and residence time at the ultimate temperature were varied to obtain different molecular weight PCSs. Alkoxides/acetylacetonates of different metals like Ti, Al, Zr etc. have been used to synthesize metal containing polycarbosilanes. The PDMS was characterized by FT-IR, TGA and DSC. FT-IR shows characteristic absorptions of Si-CH3 and Si-Si. Thermal analysis data shows a definitive characteristics degradation pattern in a step wise manner, where decomposition onsets at ~210?C and ceramization finishes at ~650?C. The PCS was characterized using FT-IR, DSC, TGA, GPC and Rheometry. The PCS showed characteristic absorptions in FT-IR for Si-H at 2100 cm-1, Si-CH3 at 1250 cm-1 and Si-CH2-Si at 1350 cm-1. PCS is a meltable and soluble solid and its softening temperature has been tuned between 90 to 180?C by carefully controlling the process conditions. ...

Topic: 9 - Polymer Derived Ceramics and Composites (incl. Reinforced foams)
Abstract no. 2227

# Dielectric property and interfacial polarization of polymer-derived amorphous silicon carbonitride

Presenting author: Prof CAO YEJIE
Affiliation: Northwestern Polytechnical University
No. 127,W. YOUXI ROAD,710072 XI'AN (SHAANXI, China)
Co-authors: WANG Yiguang MA Baisheng LI Xuqi

Polymer-derived ceramics (PDCs) are a class of advanced ceramics synthesized by the thermal decomposition of polymeric precursors, To promote the applications of PDCs in high-temperature sensors, the basic electrical properties should be understood. In the past decades, the conductivity (both DC and AC), electronic structure, semiconducting behaviors , and piezoresistivity have been fully studied. However, the dielectric behavior of PDCs has been rarely
reported. The dielectric behavior will provide useful information about the material for many applications such as wireless sensors; it will also provide more structural information about heterogeneous systems such as PDCs.
In this report, the dielectric response of polymer-derived amorphous SiCN will be discussed as a function of frequency and temperature. We show that the interfacial polarization is the major contribution to the dielectric constant at low frequencies. The interfacial polarization increases at elevated temperatures. In addition, the dielectric properties of SiCN down to 1 mHz were also characterized, and three processes were identified from the frequency dependence of the real part of permittivity.

Topic: 9 - Polymer Derived Ceramics and Composites (incl. Reinforced foams)
Abstract no. 2217

# Electromagnetic properties of Si-C-N based ceramics and composites fabricated by PDC route

Presenting author: Prof YIN Xiaowei
Affiliation: Northwestern Polytechnical University
YouYi XiLu No. 127,710072 Xi'an (Shaanxi, China)

Co-authors:

Si3N4 and SiC ceramics possess not only excellent mechanical properties but also attractive electromagnetic wave (EM) response characteristics, which make them promising materials in various applications relating to EM radiations. Owing to the tunable EM properties, the Si-C-N ceramics based on Si3N4 and SiC are attracting extensive interest in recent years. Si-C-N based materials and structure with EM properties ranging from transparent to shielding offer a high potential for light-weight, wide bandwidth, and multifunctional EM devices. Various processes have been developed to fabricate the above structural/functional ceramics. Polymer derived ceramics (PDC) route not only has the potential to in-situ form the new type ceramics, but also make it possible to realize the ceramics-based hybrid structure at lower temperature. The key developments and future challenges in this field are summarized. The main issues regarding permittivity of Si-C-N ceramics and composites are discussed, with an emphasis on the EM transmission, shielding and absorption mechanisms that are responsible for EM properties.

# Topic: 10

# Carbon/carbon composites

Topic: 10 - Carbon/carbon composites
Abstract no. 1976

# Resistant Performance of Atomic Oxygen Damage on ZrC Component Modified C/C Composite


Presenting author: Dr LIU Guanghai
Affiliation: Northwestern Polytechnical University
Youyi Road 127#,Beilin District,710072 Xi'an (Shaanxi Province, China)

Co-authors: LAIFEI Cheng (Northwestern Polytechnical University)



For the sake of improving high energy atomic oxygen(AO) resistant abilities of C/C composite, ZrC component, as an AO resistant modification, was induced and investigated to be compared with C/C in this research. The degradation and radiation resistant mechanisms were focused on by using method of ground environmental simulation test. Both of ZrC modified C/C and C/C samples were exposed to 5eV energetic atomic oxygen, such setting of AO energy simulates the space environment of low Earth orbit(LEO). ZrC modification improvements of AO resistance ability were ascertained by providing a series tests of bending strength, SEM(scanning electron microscopy), XPS(X-ray photoelectron spectroscopy), EDS and quality changes.

Topic: 10 - Carbon/carbon composites
Abstract no. 2006

# Effect of the temperature on the mechanical behaviour and the damage of carbon-carbon composite

Presenting author: M VOIRIN Thibaut
Affiliation: University of Lyon, INSA Lyon, UCBL, MATEIS
345 Avenue Gaston Berger,69621 Villeurbanne (France)

Co-authors: REYNAUD Pascal (Univ Lyon / INSA Lyon / MATEIS), FANTOZZI Gilbert (Univ Lyon / INSA Lyon / MATEIS)

Carbon-carbon composites are very interesting materials, considering their low weight, good mechanical properties and stability at high temperature.
The main objective of this work is to improve the understanding of the carbon-carbon composite materials, used under original loading modes (under compressive stress, inter-laminar shear stress, etc.) and their evolution with the increase of temperature.
In this paper, the compressive behaviour in Z direction and XY behaviour at room temperature and high temperature are studied. This study shows that the mechanical properties are enhanced when the temperature increases. This evolution is attributed to changes in the crushing and delamination of the plies.
Damaged materials are also observed post-mortem using optical microscopy and Scanning Electron Microscopy in order to better understand the effects of constituents evolutions and their consequences on the mechanical behaviour of the composite.

Topic: 10 - Carbon/carbon composites
Abstract no. 2134

# The ablation behaviors and mechanism of C/C-SiC-ZrC composite by $CO_2$ laser

Presenting author: Dr GENG Li
Affiliation: Northwestern Polytechnical University
Youyi road, number 127,710072 Xi'an (ShaanXi, China)

Co-authors:

C/C-SiC-ZrC composite was prepared by chemical vapor infiltration (CVI) combined with $Si_{0.87}$-$Zr_{0.13}$ alloyed reactive melt infiltration (RMI). The ablation behaviors of the composite were investigated by high energy $CO_2$ laser under Argon atmosphere. The depths and 3D profiles of laser ablation holes were measured by Laser Confocal Microscope (LCM). The temperature distribution was simulated by a 3D finite element model through COMSOL Multiphysics 5.4. The surface of the composites which ablated for 5s and 100s was discussed by different regions respectively. The ablation mechanism and morphology evolution were discussed according to the analysis result of the SEM and EDS. It was found that C/C-SiC-ZrC has a lowest ablation rate than C/C and C/C-SiC. C-SiC–ZrC matrix has a gradient structure, ZrC was enriched on the sample surface, which has a positive effect in improving the anti-ablation performance of the composite.

Topic: 10 - Carbon/carbon composites
Abstract no. 2127

# Damage monitoring of a 3D C/C composite by means of acoustic emission

Presenting author: Dr EL YAGOUBI JALAL
Affiliation: Institut de Mécanique et d'ingénierie
Université de Bordeaux,351 cours de la libération,33405 TALENCE Cedex (France)
Co-authors: EL YAGOUBI Jalal (Université de Bordeaux UMR 5295 Institut de Mécanique et d'Ingénierie (I2M), Bordeaux F-33000 France), TOSON Bernard (CEA-CESTA Le Barp F-33114 France), HALLO Ludovic (CEA-CESTA Le Barp F-33114 France)

This paper deals with the detection and monitoring of damage formation and propagation in the bulk of a three-dimensional carbon/carbon composite (3D C/C). At the initial state the existing cracking network may be quite extended due to the manufacturing process. In addition during loading, the composite develops cracks, that will affect the mechanical behavior, and fiber failures that lead to the fatal rupture. The objective of this work is to investigate the damage scenario by using acoustic emission diagnostic. For this purpose, load-unload-reload tensile and modified V notched shear tests have been conducted. Those tests have been simultaneously monitored with the Digital Image Correlation (DIC) and the acoustic emission (AE) techniques. The full-field displacement mappings have been obtained by DIC (2D) and the typical non-linear stress-strain curves have been plotted. We show that AE provides accurate location of damage based on the density events location maps. This finding is already well known for homogeneous material but apparently new for 3D C/C. Moreover different stages of damage formation have been identified based on the AE activity and AE signals parameters. A significant increase in AE energy has been observed close to the final failure of the specimens. The number of detected AE events is more important with modified V notched shear tests in comparison with tensile tests. K-means clustering associated with principal component analysis have been also used to discriminate between failure mechanisms. This analysis is combined with X-ray tomography observations to explore the crack network that develops during loading.

Topic: 10 - Carbon/carbon composites
Abstract no. 2103

# Tailoring the interlaminar stress of laminated carbon/carbon composites towards improved strength and reliability

Presenting author: Dr SHEN Qingliang
Affiliation: Northwestern Polytechnical University
127 West Youyi Road, 710072,710072 Xi'an (Shanxi, China)

Co-authors:

Carbon/carbon composites are critical structural materials that are widely applied at high temperatures and the reliability of the composites is of primary concern. The present work describes a feasible method to simultaneously improve the strength and decrease the strength deviation by optimizing the stacking sequence of the laminate. An alternatingly stacking of low texture ply and high texture ply can induce a compressive residual thermal stress at the mesoscale in the high texture ply due to the mismatch of thermal expansion between them. This residual stress facilitates the deflection and arresting of the delamination crack and decrease the sensitivity of the composite strength to the flaws. From the engineering aspect, this method provides a meaningful strategy to decrease the weight of carbon/carbon components while remain its reliability, which is of great importance in reducing the cost during the design and fabrication period for aerospace applications.

Topic: 10 - Carbon/carbon composites
Abstract no. 2009

# Questions on the multiscale study of the mechanical and thermomechanical behavior of C/C composites


Presenting author: M VIGNOLES Gerard L.
Affiliation: Univ. Bordeaux - LCTS
3 Allée La Boétie,33600 PESSAC (Aquitaine, France)

Co-authors: COUéGNAT Guillaume (CNRS - LCTS), LEYSSALE Jean-Marc (CNRS - ISM Bordeaux), FARBOS Baptiste (Université de Bordeaux - LCTS), GILLARD Adrien (Université de Bordeaux - CEA - LCTS), RAUDE Amandine (Université de Bordeaux - LCTS), CHARRON Morgan (Université de Bordeaux - LCTS), JOUANNIGOT Stéphane (CNRS - LCTS), CATY Olivier (Université de Bordeaux - LCTS)



The mechanical and thermomechanical behavior of C/C composites, although well known since decades, poses some fundamental questions that are still open.

- why is the elastic modulus of pyrocarbon matrices only 10% of the graphene value? We build hints to the answer on the basis of molecular dynamics simulations and on orientation distributions of the anisotropy axes in polycrystalline graphite.
- what is the role of internal interfaces in the non-linear behavior of C/C composites? We present a modeling approach supported by push-out tests to address this issue.
- how to measure the heat expansion of pyrolytic carbons? We propose an experimental determination method and show some results.
- by what material parameters is the heat expansion of C/C composites primarily controlled on the macroscale? We show that answering this question is linked to the previous issue of the non-linear mechanical response.

Topic: 10 - Carbon/carbon composites
Abstract no. 2082

# Ablation behavior of carbon/carbon composites doped by refractory metal carbides under extreme environments


Presenting author: M LI KEZHI
Affiliation: Northwestern Polytechnical University
N0.127 West Road, Xi'an China,710072 Xi'an (China)

Co-authors:



Wedge-shaped samples of ZrC-SiC inhibited carbon/carbon (C/C-ZrC-SiC) composites fabricated by reactive melt infiltration (RMI) were tested on an oxyacetylene torch with the heat flux of 2.38 MW/m2 for 120 s. The reaction-bonded ZrC-SiC ceramics were developed in the fiber webs of the porous C/C preform after RMI of ZrSi powder. C/C-ZrC-SiC sample underwent a high temperature gradient and tip temperature was as high as 2485 ºC during steady state ablation stage. The tip temperature of carbon/carbon wedge-shaped sample was maintained at 1735 ºC. The ablation resistance performance of C/C-ZrC-SiC was significantly superior to that of C/C and no spallation occurred on the tip of C/C-ZrC-SiC after ablation. Microstructure characterization revealed a porous ZrO2 forming at the tip of C/C-ZrC-SiC sample due to the complete depletion of SiC, carbon matrix and carbon fibers in ultrahigh temperature conditions. The formation of ZrO2 at the tip region was capable of playing a thermal barrier, enduring and resisting the high pressure and mechanical denudation of the flame stream. The mechanical denudation of the flame stream acting on the sample surface was weakened by the blocking of the tip and the surface was mainly affected by the ultrahigh temperature oxidation. Microstructure evolution on the sample surface orderly revealed a porous ZrO2 layer at the region near the tip, layered structure of ZrO2 outer layer with a dense ZrO2-SiO2 sub layer at the middle region as well as SiO2 layer at the tail region due to presence of temperature gradient. Particularly, at the middle region, grain size of ZrO2 in ZrO2-SiO2 sub layer was smaller than that in the outer layer due to retention of SiO2 glass. Thermal chemical erosion was alleviated by the thermal barrier role of the outer layer ZrO2 combined with the oxygen diffusion barrier of the sub layer ZrO2-SiO2.

Topic: 10 - Carbon/carbon composites
Abstract no. 2446

# Ablation behavior of carbon/carbon composite modified by novel solid solution boride-carbide


Presenting author: M ZENG Yi

Affiliation: State Key Laboratory of Powder Metallurgy, Central South University, Changsha 410083, PR China
932 South Lushan Rd,Changsha Hunan 410083 P.R. China,410083 Changsha (Hunan, China)

Co-authors: YE Ziming (State Key Laboratory of Powder Metallurgy Central South University Changsha 410083 China), XIONG Xiang (State Key Laboratory of Powder Metallurgy Central South University Changsha 410083 China), WANG Yalei (State Key Laboratory of Powder Metallurgy Central South University Changsha 410083 China), CHEN Zhaoke (State Key Laboratory of Powder Metallurgy Central South University Changsha 410083 China), SUN Wei (State Key Laboratory of Powder Metallurgy Central South University Changsha 410083 PR China), LIN Wang (State Key Laboratory of Powder Metallurgy Central South University Changsha 410083 PR China)



A novel solid solution boride-carbide modified carbon/carbon(C/C) composite were prepared via reactive melt infiltration and pack cementation, Hf-Zr-Ti-C solid solution carbide was firstly introduced into the C/C composite by reactive melt infiltration, followed by Hf-Zr-Ti-B-Cand SiC coating on to the surface through pack cementation. The phase assemblage, morphology and chemical composition of the prepared boride-carbide-C/C composite was studied, the evolution of constituent, morphology and microstructure of the composites during ablation is investigated and the ablation mechanism is presented. The composite displays significantly improved ablation resistance at temperatures of 2500?. The good ablation resistance is mainly attributed to a dense and stable multi-oxide scale composed of solid solution oxide grain skeleton with borosilicate glass phase sealing the pores and defects and the unique intermediate interface of Hf-Zr-Ti-O-C formed between the oxides and carbide during the ablation test. The solid solution oxide grain skeleton and borosilicate glass phase act as a relatively impervious barrier as well as a thermal barrier while the interface composed of oxide nanocrystalline and amorphous carbon, which can effectively retard the diffusion of oxygen from the oxide layer to the carbide, leading to a desirable oxidation resistance. The results obtained in this work allow us to consider this novel boride-carbide-C/C composite as a very promising candidate for extreme applications.

Topic: 10 - Carbon/carbon composites
Abstract no. 2173

# Experimental Research on the Mechanical and Thermal Behavior of Carbon/Carbon Sandwich Panels for Ultra Stable Structure

Presenting author: M LIU Yufeng
Affiliation: National key laboratory of advanced functional composite materials, Aerospace research institute of materials and processing technology
South dahongmen Road 1th,Daxing district,100076 Beijing (Beijing, China)
Co-authors: ZHANG Zhongwei LI Tongqi XU Zhenghui WANG Jinmin

The electrostatic gravity gradiometer is the main payload of gravity gradient satellite platform. To meet the outstanding measurement requirements, the electrostatic gravity gradiometer structure must provide high dimensional stability under the expected environmental conditions. Carbon/carbon technology has been selected for the accelerometers and their electronics. This technology offers good thermoelastic characteristics, high panel stiffness-to-mass ratio and low sensitivity to moisture. The purpose of this paper is to present the evaluation of a new kind of carbon/carbon sandwich panels with the aim to make ultra stable structures that could offer high level of stability requested by space science high performances instruments. Carbon/carbon skins and carbon/carbon honeycomb were prepared by chemical vapor deposition and organic joining technology was used to bond carbon/carbon sandwich. Mechanical behaviors of the sandwich panels have been analyzed by out-of-plane compression, in-plane compression and three point bending experiments. CT was used for testing the application conditions of panels' inner quality. Thermal behaviors of the panels have been analyzed by thermal conductivity and expansion tests. The results indicated that the carbon/carbon sandwich have a very low thermoelastic sensitivity (In plane coefficient of the thermal expansion?5×10-7) and the mechanical properties meet the design requirements.

Topic: 10 - Carbon/carbon composites
Abstract no. 1962

# Effect of fatigue on damping behavior and flexural strength of C/C composites


Presenting author: M SUN Guodong
Affiliation: Chang'an University
Middle-section of Nan'er Huan Road,710064 Xi'an (Shaanxi, China)

Co-authors: LV Wenjiong (Chang'an University), LI Hui (Chang'an University), YU Pengfei (Chang'an University), WANG Zhenjun (Chang'an University)



C/C(carbon/carbon) composites have excellent properties such as low density, high temperature resistance and ablation resistance, etc. As a kind of high temperature thermal structure material, they have been widely used in the fields of aviation, spaceflight and so on. In order to master the evolution of mechanical and structural properties of C/C composites due to alternating loads during the service process, laminated C/C composites were fabricated by precursor infiltration pyrolysis (PIP) method, and the influences of tensile-tensile fatigue load on damping behavior and flexural strength of C/C composites were studies. The results showed that fatigue made a similar effect on damping behavior and mechanical properties. The damping and flexural strength of C/C composites both increased after fatigue, which was independent of loading stress levels and loading cycles. The fatigue loads made the strong-bonding fiber/matrix interface weaker, which was conducive to the formation of fiber debonding, pulling-out and interfacial sliding. These microstructure changes were beneficial to the damping and flexural strength of C/C composites.

Topic: 10 - Carbon/carbon composites
Abstract no. 2255

# Tensile strength and creep behavior of carbon-carbon composites at elevated temperature

Presenting author: Dr GOTO Ken
Affiliation: Institute of Space and Astronautical Science, Japan Aerospace Exploration Agency
3-1-1, Yoshinodai, Chuo-ku,229-5210 Sagamihara (Kanagawa, Japan)

Co-authors:

Tensile strength and creep behavior of carbon-carbon composite (C/C) reinforced with a high modulus type PAN based carbon fiber (C/C(M40)) and C/C reinforced with a low modulus type PAN based carbon fiber (C/C(T300)) were examined from room temperature to 2773K in an inert atmosphere. Tensile strength of both C/Cs at elevated temperature up to 2273 K. Over 2273 K, tensile strength of C/C(M40) continued to increase, on the other hand, tensile strength of C/C(T300) degraded. Tensile strength of C/C(T300) could not obtained at 2773 K due to the decline of applied stresss during tensile test by rapid creep deformation. Creep deformation appeared from 1873 K in both C/Cs regardless to the type of the reinforcing carbon fiber. No variation was found in the activation energy, however, C/C reinforced with higher modulus carbon fiber exhibited lower stress exponent. The creep resistance of C/Cs could be enhanced by a carbon fiber processed at higher heat treatment temperature. The heat treatment of C/C itself also enhanced creep resistance, however, its effect could not reach to that from heat treatment of carbon fiber. Strength enhancement of C/C(M40) by creep deformation was investing further by tensile strength difference at room temperature by creep deformation. Tensile strength of carbon fiber after creep deformation was also examined to find out ruling mechanism. From the result, strength enhancement of C/Cs at high temperature was achieved by fracture strain enhancement derived from improvement fracture toughness of carbon fiber at elevated temperature. Creep deformation less than 5 % enhanced tensile strength of C/Cs. The enhancement by creep deformation was mainly caused by the enhancement of Young's modulus of carbon fiber.

Topic: 10 - Carbon/carbon composites
Abstract no. 2158

# Effect of different curvature radius on preparation and ablation resistance of ZrC-SiC/SiC coating on sharp-shape C/C composites


Presenting author: Dr LI Bo

Affiliation: State Key Laboratory of Solidification Processing, Carbon/Carbon Composites Research Center, Northwestern Polytechnical University
No. 127, Youyi Road(West),Northwestern Polytechnical Univ,710072 Xi'an (Shaanxi, China)

Co-authors: LI Hejun (State Key Laboratory of Solidification Processing Carbon/Carbon Composites Research Center Northwestern Polytechnical University)



To improve the ablation resistance of carbon/carbon (C/C) composites, the ZrC-SiC coating using in-situ reaction method was prepared on SiC-coated C/C composites, which were machined into sharp leading edge (SLE) shape to simulate the real working condition of leading edges of hypersonic vehicles. The effect of different curvature radius (R) of the SLEs on the preparation and ablation resistance of the coatings were studied. The R value was 0.5 (R1), 1 (R2), 2 (R3), 3mm (R4), respectively. With increasing R value, the cracking patterns of the coatings were changed from wide and deep cracks concentrated on the top of the wedges to shallow and narrow cracks uniformly distributed, meanwhile the number of cracks increased. The coating thickness and the distribution of the ZrC and SiC phases in the coatings were influenced by R value slightly. During 60s of oxyacetylene ablation, SLEs with low R values experienced more severe ablation. The maximum surface temperature were 2226? (R1), 2171? (R2), 2116? (R3), 2090? (R4). With increasing R value, the mass ablation rate increased whereas the line ablation rate decreased. Finite element analysis was used to simulate the surface temperature and shear stress distribution during ablation of different SLEs. The simulated cloud images were in agreement with the experimental results, which can assist to explain the ablation behavior. Combined with the microstructure, the ablation behavior of R2 as an example was analyzed in detail. In the center ablation region, the temperature was highest, leading to the damage of coating. The top and side of the wedge were subjected to stagnation ablation and shear ablation, respectively, causing the different morphologies of the brim ablation regions on top and side. It is molten SiO2 with porosities on the top while a large amount of SiO2 bubbles on the side.

Topic: 10 - Carbon/carbon composites
Abstract no. 2449

# Effect of the deposition conditions on the microstructure of pyrolytic carbon and densification of C/C composites fabricated by ICVI


Presenting author: Prof WANG Yalei

Affiliation: State Key Laboratory of Powder Metallurgy, Central South University, Changsha 410083, China
932 South Lushan Rd,Changsha Hunan 410083 P.R. China,410083 Changsha (Hunan, China)

Co-authors: FANG Chong (State Key Laboratory of Powder Metallurgy Central South University Changsha 410083 China), XIONG Xiang (State Key Laboratory of Powder Metallurgy Central South University Changsha 410083 China), TANG Zhonghua (State Key Laboratory of Powder Metallurgy Central South University Changsha 410083 China), CHEN Zhaoke (State Key Laboratory of Powder Metallurgy Central South University Changsha 410083 China), SUN Wei (State Key Laboratory of Powder Metallurgy Central South University Changsha 410083 China), ZENG Yi (State Key Laboratory of Powder Metallurgy Central South University Changsha 410083 China), WANG Lin (State Key Laboratory of Powder Metallurgy Central South University Changsha 410083 China)



The effect of the deposition temperature and precursor ratio on the densification of C/C composites and microstructure of pyrolytic carbon (PyC) has been studied by polarized light microscopy (PLM), scanning electron microscopy (SEM), Raman spectroscopy, X-ray diffraction (XRD) and transmission electron microscopy (TEM) combined with selected area electron diffraction (SAED). C/C composites were produced by isothermal, isobaric chemical vapor infiltration at different deposition temperature with methane and methane/propylene mixtures at different ratio as precursor. Deposition rates as well as matrix microstructure controlled by the deposition temperature and precursor ratio differ with temperature and precursor ratio. Under the optimal condition, an entirely rough laminar pyrolytic carbon matrix and C/C composites with the bulk density of 1.52 g/cm3 are obtained after 150 h deposition. Furthermore the largest density distribution discrepancy is 0.12 g/cm3 and the lowest density zone is found to be at or near the middle.

Topic: 10 - Carbon/carbon composites
Abstract no. 2132

# Microstructural evolution of high texture and medium texture pyrolytic carbon at different temperatures

Presenting author: Mme WANG Tiyuan
Affiliation: Northwestern Polytechnical University
127 West Youyi Road,710072 Xi'an (China)

Co-authors:

The difference of microstructural evolution of high texture (HT) and medium texture (MT) pyrolytic carbon (PyC) at different temperatures was investigated by polarized light microscopy (PLM), scanning electronic microscopy (SEM) and transmission electron microscopy (TEM). The results demonstrated that the delamination of MT and HT was different after heat treatment. HT matrix was divided into many nano-thickness sublayers which are accompanied by multiple microcracks, while MT matrix was divided into layers of micron-thickness firstly after heat treatment, there are many microcracks and nano-scale sublayers in these layers. The upward tendency of the lattice parameters (La and Lc) of HT exceeds MT, while the contact region width in HT is always narrower than that in MT, and there are a large number of nanopores after 2450 °C. The micro-indentation results showed that the indentation depth of HT was deeper than that of MT, the micro-hardness of MT treated was greater than that of HT, and the regularity of elastic modulus is same to hardness. The micromechanical difference also verifies the microstructure differences.

# Topic: 11

# Thermomechanical behavior and performance of Composites

Topic: 11 - Thermomechanical behavior and performance of Composites
Abstract no. 2219

# New Strategies to Understand Oxidation Processes in Heterogeneous, Non-oxide Ceramic Composites


Presenting author: Prof POERSCHKE David
Affiliation: Unniversity of Minnesota
421 Washington Ave SE,55455 Minneapolis (Minnesota, United States)

Co-authors: VERRIJT Koen (University of Minnesota)



The implementation of SiC-based ceramic composites has been limited, in part, by challenges processing high purity, defect-free matrices, and the resultant effects on their performance in extreme environments. Whereas dense SiC or Si3N4 matrices effectively prevent the oxidant ingress from the service environment, matrix heterogeneity including spatial variations in porosity, composition, crystallinity, and grain size can accelerate oxidant ingress and lead to premature component failure. This is particularly true in composites produced using polymer derived ceramics, wherein the final matrix may be amorphous or semi-crystalline, and typically contains pores and microcracks ranging from nanometers to tens of microns. Elucidating the oxidation mechanisms in these materials is necessary to model environmentally-induced CMC strength degradation. However, experimental investigations of oxidant transport are complicated by the interplay between surface and internal oxidation processes. Oxygen ingress can occur by gaseous transport through open porosity, by grain boundary transport in the nanocrystalline structure (particularly when the grain boundaries are decorated with free carbon or amorphous material), and in the solid state through the amorphous PDC phases. This presentation will describe recent efforts to develop new experimental methodologies to study oxidation processes in heterogeneous, Si-based ceramics. The effort is developing novel approaches using solid state oxygen pumps to achieve precise control over the local oxygen chemical potential and enable pump-probe experiments to extract oxidation kinetics. In combination with experiments performed on specially-designed specimens featuring internal chemical markers to visualize the advancing oxidation fronts, the insights shed new light on the factors controlling oxidation behavior and enable the design of new materials with improved performance.

Topic: 11 - Thermomechanical behavior and performance of Composites
Abstract no. 2046

# Microstructure-based modeling of the thermomechanical properties of ceramic matrix composites


Presenting author: Dr MAILLET Emmanuel
Affiliation: GE Research
1 Research Circle,12309 Niskayuna (NY, United States)

Co-authors: MOSCINSKI Michael (GE Research), BAGRI Akbar (GE Research), MEYER Pascal (GE Research), DUNN Daniel (GE Research)



A fundamental requirement for designing mechanical components with ceramic matrix composites (CMCs) is understanding their thermomechanical properties. This is usually achieved by extensive testing and characterization. The present approach aims at developing models to quantify the effects constituent and microstructure variations have on the thermomechanical behavior of CMCs. The properties of interest include thermal-elastic, ultimate tensile strength and interlaminar strength. This presentation will discuss the generation of representative modeled microstructures, modeling frameworks, effects of key constituent properties and microstructural features, as well as validation with experimental data on known microstructures.

Topic: 11 - Thermomechanical behavior and performance of Composites
Abstract no. 2185

# Life Prediction of SiC/SiC Ceramic Matrix Composites for Turbine Applications

Presenting author: Dr SUDRE Olivier
Affiliation: Pratt & Whitney
---,--- --- (---, United States)

Co-authors: COX Brian N. (Arachne Consulting), YANG Qingda D. (University of Miami), PARTHASARATHY Triplicane (UES inc), MARSHALL David B. (University of Colorado), VINUP Michael (Honeywell Aerospace), KUMAR Brijesh (Honeywell Aerospace), KRUK Natalie (Honeywell Aerospace)

The development of ceramic matrix composites heavily relies on the premise that cracks can be managed during the lifetime of a component in service. These cracks are the pathways for the environment to degrade the fiber tows further enabling the damage progression within the structure. We are currently developing a suite of high-fidelity, physics based models to predict the life of ceramic matrix composites under jet engine environments. Our approach follows the Integrated Computational Materials Engineering (ICME) precepts by creating virtual specimens and sub-components for the calibration and the validation of the models. The high-fidelity computational model is based on the Binary Model, a computationally efficient and adaptable finite element methodology where individual tows of the coupon or component are individually introduced in the finite element mesh. The code tracks the evolution of the matrix degradation as either continuum damage or discrete microcracks in a sub-component-scale entity possessing a heterogeneous microstructure. Additionally, the code follows the evolution of the fiber tow properties based on position, load and local environment within the structure via an independent subroutine that takes into account the various oxidation mechanisms of the SiC matrix, BN interface and Hi-Nicalon fiber. The subroutine was independently calibrated using stress-rupture test data on fiber bundles. The material studied was a commercial 2D SiC/SiC composite in the form of flat tensile test coupons for calibration and extracted C-shaped coupons from a vane sub-elements tested in an engine environment for validation.

Topic: 11 - Thermomechanical behavior and performance of Composites
Abstract no. 1929

# Microstructure and damage evolution of SiCf/PyC/SiC and SiCf/BN/SiC mini-composites: A synchrotron X-ray computed microtomography study


Presenting author: Prof YANG Haitang
Affiliation: Central South University
,No. 932 Lushan South Street,410083 Changsha (Hunan Province, China)

Co-authors:



SiCf/PyC/SiC and SiCf/BN/SiC mini-composites comprising single tow SiC fibre-reinforced SiC with chemical vapor deposited PyC or BN interface layers are fabricated. The microstructure evolutions of the mini-composite samples as the oxidation temperature increases (oxidation at 1000, 1200, 1400, and 1600 °C in air for two hours) are observed by scanning electron microscopy, energy dispersive spectrometry, and X-ray diffraction characterization methods. The damage evolution for each component of the as-fabricated SiCf/SiC composites (SiC fibre, PyC/BN interface, SiC matrix, and mesophase) is mapped as a three-dimensional (3D) image and quantified with X-ray computed tomography (as shown in Figs. 1-4). The mechanical performance of the composites is investigated via tensile tests.

The results reveal that tensile failure occurs after the delamination and fibre pull-out in the SiCf/PyC/SiC composites due to the volatilization of the PyC interface at high temperatures in the air environment. Meanwhile, the gaps between the fibres and matrix lead to rapid oxidation and crack propagation from the SiC matrix to SiC fibre, resulting in the failure of the SiCf/PyC/SiC composites as the oxidation temperature increases to 1600 °C. On the other hand, the oxidation products of B2O3 molten compounds (reacted from the BN interface) fill up the fracture, cracks, and voids in the SiC matrix, providing excellent strength retention at elevated oxidation temperatures. Moreover, under the protection of B2O3, the SiCf/BN/SiC mini-composites show a nearly intact microstructure of the SiC fibre, a low void growth rate from the matrix to fibre, and inhibition of new void formation and the SiO2 grain growth from room to high temperatures. This work provides guidance for predicting the service life of SiCf/PyC/SiC and SiCf/BN/SiC composite materials, and is fundamental for establishing multiscale damage models on a local scale.

Topic: 11 - Thermomechanical behavior and performance of Composites
Abstract no. 1982

# Study of damage of complex weaved CMC structures submitted to multi-axial thermo-mechanical loading by an in-situ experimental approach

Presenting author: M TURPIN Léonard
Affiliation: LCTS UMR5801
3 allée de la Boétie,33600 Pessac (France)

Co-authors:

SiC/SiC Ceramic Matrix Composites (CMCs) are promising candidates to replace super-alloys in aircraft engines to reduce weight and increase the operating temperature hence to improve engine efficiency. SiC/SiC CMCs are made up of both silicon carbide weaved preform and matrix. Plain weave is optimized both for its complex geometry and loading it is subjected to.
Here, the aim is to understand the link between micro-structure and local thermo-mechanical behaviour to define a multi-axial first-damage criterion. This behaviour depends on the weaved pattern, its singularity and matrix quality. It requires to observe, at a fine spatial resolution, areas which are hardly accessible. Thus, an in-situ approach, using tomography has been chosen to assess damage. Integrating Digital Volume Correlation (DVC) to an image-based mechanical model, faithful to the true micro-structure, provides the displacement field of both the studied volume and the identified thermo-mechanical loading.
High temperature (1,200°C) tensile in-situ tests on regular SiC/SiC structures have already demonstrated the benefit of tomography and DVC association to understand the material behaviour. A generalisation of this approach is presented here.

Topic: 11 - Thermomechanical behavior and performance of Composites
Abstract no. 1928

# Multi-Axial Failure Characterization and Modeling of Oxide-Oxide CMCs


Presenting author: Dr JEFFERSON George
Affiliation: AFRL, Materials Directorate
2230 Creekview Pl,45305 Bellbrook (OH, United States)

Co-authors: PRZYBYLA Craig (AFRL Materials Directorate), ZAWADA Larry (Universal Technologies Corp), PIERCE Jennifer (University of Dayton Research Institute)



Oxide-Oxide CMCs were tested under various ratios of tension and in-plane shear stress using a tension-torsion tube test. Specimens were fabricated using an involute layup, and successfully tested under proportionally loaded tension-torsion, and compression-torsion. Phenomenological PMC-based models were investigated for describing the failure envelope and were found to be suitable with minor modification. The composite behavior was further investigated subject to non-proportional and cyclic loading. The results are used to validate and calibrate a continuum damage model for CMCs using a laminate based finite element model of the involute tube. Additionally a discrete binary fiber-matrix model was developed to evaluate the applicability of matrix level continuum damage models.

Topic: 11 - Thermomechanical behavior and performance of Composites
Abstract no. 1946

# Fatigue Characterization of Melt Infiltrated SiC/SiC Ceramic Matrix Composites under Burner rig Conditions

Presenting author: M PANAKARAJUPALLY Ragavendra Prasad
Affiliation: The University of Akron
302 buchtel commons,sumner street,44325 Akron (Ohio, United States)
Co-authors: K Manigandan (The University of Akron), MORSCHER Gregory (The University of Akron)

Future gas turbines are aimed for higher efficiency with lower exhaust emissions. An increase in turbine inlet temperature (TIT) increases the efficiency of the jet engine. Ceramic Matrix Composites (CMC's) are the candidate materials because of their high temperature capability, low weight and high specific strength. These materials are prone to oxidation followed by surface recession under the oxidative water vapor environment which limits their operation. One has to test and characterize these CMC's in similar jet engine conditions namely thermal gradient stress, velocity and water vapor for the successful implementation in jet engines. For this reason, a unique experimental facility (Burner rig) is built which can simulate combined mechanical and combustion loading. In this study, melt infiltrated (MI) SiC/SiC CMC's are investigated under the burner rig conditions to characterize and study the effect of combustion environment on the life and mechanical properties. Several tests are performed under tension-tension fatigue condition at different stress levels at a frequency of 1 Hz, stress ratio of 0.1 and a specimen surface temperature of 1200° C. NDE techniques such as electrical resistance (ER) is used as in-situ health monitoring technique and two forward lean infrared cameras (FLIR) are used to monitor the specimen surface temperature. Similar tests are performed in isothermal static furnace for comparison. Post test analysis are performed on the fracture surfaces using scanning electron microscope to understand the damage mechanisms and the extent of embrittlement.

Topic: 11 - Thermomechanical behavior and performance of Composites
Abstract no. 1772

# Characterization of the damage process of C/SiC ceramic matrix composites by various monitoring techniques

Presenting author: M GOULMY Jean-Patrick
Affiliation: Laboratoire des Composites ThermoStructuraux (LCTS), Université de Bordeaux et CNRS, Safran, CEA
3 allée de la Boétie,33600 Pessac (France)

Co-authors: ALLEMAND Alexandre (CEA Le Ripault), CAMUS Gerald (Laboratoire des Composites ThermoStructuraux (LCTS), Université de Bordeaux et CNRS Safran CEA), REBILLAT Francis (Laboratoire des Composites ThermoStructuraux (LCTS), Université de Bordeaux et CNRS Safran CEA)

Mechanical properties of C/SiC composites are mainly a function of the matrix manufacturing process, C fibers used and the architecture of the fibers preform. The objective of this work is to relate the microstructure and mechanical behaviour of C/SiC composites of different origins, tested under several conditions. For this purpose, a microstructural characterization has been carried out using Optical Microscopy, Scanning Electron Microscope and X-ray Diffraction. The microstructural identifications helped to understand the differences in mechanical behaviour observed between the materials and to make the link with the different processing techniques. Mechanical tests were carried out at room temperature and high temperatures with damage monitoring (acoustic emission, electrical resistivity and interposed unloading/reloading cycles).
The objective of this talk will be to present and discuss the used approach and the results thus obtained. Three materials, developed using the Chemical Vapour Infiltration and Melt Infiltration processes, will be compared from a microstructural and behavioural point of view.

Topic: 11 - Thermomechanical behavior and performance of Composites
Abstract no. 2229

# Fabrication of artificial defects and their effect on the mechanical properties of C/C-SiC


Presenting author: M HAUSHERR Jan Marcel
Affiliation: Fraunhofer Center for High Temperature Materials and Design
Gottlieb keim Str. 62,95448 Bayreuth (Germany)

Co-authors: RüDINGER Arne (Fraunhofer Center for High Temperature Materials and Design), GORYWODA Marek (Hof University), WAGNER Lukas (Center for high Temperature Materials and Design / Hof University)



Determining the effect of defects in fiber-reinforced materials, such as polymer matrix composites (PMCs) can be studied by creating artificial flaws in these materials, such as including PTFE foil for delaminations. For fiber-reinforced ceramics (CMCs), this approach is more difficult due to the more complicated production routes of CMCs, which involve several steps that include processing at high temperatures. This work deals with the purpose of fabricating defects into carbon fiber reinforced silicon carbide (C/C-SiC) composites in such a way that they can be detected by non-destructive material testing methods in and after each production step of the composite.
It was shown that the defects produced using boron nitride (BN) and alumina fiber roving were stable over the entire manufacturing process and could be detected by ultrasound and x-ray computed tomography techniques.
To determine an effect of the included defects, an initial sampling of bending and tensile samples with artificial defects was manufactured, tested and compared with defect free reference materials.
It was observed that the The bending tests showed a lower flexural strength and failure strain compared to samples with no such defects.

Topic: 11 - Thermomechanical behavior and performance of Composites
Abstract no. 1817

# Stress-environmental degradation of SiC/BN/SiC CMCs for aero-engines applications


Presenting author: Dr GAVALDA DIAZ Oriol
Affiliation: Imperial College London
Royal School of Mines,SW7 2AZ London (London, United Kingdom)

Co-authors: SAIZ Eduardo (Imperial College London), GIULIANI Finn (Imperial College London), VANDEPERRE Luc (Imperial College London), AL NASIRI Nasrin (Imperial College London), MARQUARDT Katharina (Imperial College London), GALE Louise (Rolls Royce plc), HARRIS Stephen (Rolls Royce plc), EDMONDS Ian (Rolls-Royce plc)



The need to increase the cycle efficiency and reduce NOx emissions from aero-engines has promoted the development of Ceramic Matrix Composites (CMCs), allowing a higher in-service temperature which will lead to a significant improvement in fuel consumption and weight savings. Silicon Carbide (SiC) based Ceramic Matrix Composites (CMCs) entered in service in aircraft turbine engines as replacements for some Ni-based superalloys and are currently expected to grow in demand. However, their stability in the harsh operation environments that form in the hot part of an aero-engine is still a matter of concern.
The present study focuses on the mechanisms of degradation and damage progression in Hi-Nicalon SiC/BN/SiC CMCs under the stress-environmental solicitations present in the hot part of aero-engines. This is achieved via in-depth material characterisation and mapping of the oxide products formed within the CMC architecture. Furthermore, SEM in-situ techniques are used to understand how these oxide species affect the micromechanical performance of the fibre-matrix interfaces. The goal is to understand the fundamentals governing the stress-environmental degradation of SiC-based CMCs in aero-engine environments in order to guide the design of future materials.

Topic: 11 - Thermomechanical behavior and performance of Composites
Abstract no. 1988

# Thermomechanical behaviour of 3D C/C composites: effect of shear loading on microstructure features and multiscale modelling approach

Presenting author: M HUCHET Guillaume
Affiliation: CEA DAM, Le Ripault
B.P. 16,37260 Monts (France)

Co-authors:

3D C/C composites show typical behaviour under mechanical loading at high temperature. Firstly, tensile behaviour is governed by stiff fibres tows in the direction of loading, leading to linear behaviour. Secondly, non-linearity phenomenon occurs when interfaces carry loads at different scales of the microstructure. Opening, closing and sliding displacements signify local mechanisms, sources of non-linear elasticity in compression and irreversible damage, initiating non-linearity under shear (Figure 1).
To macroscopically understand the way how microstructure is able to govern such behaviours, useful experimental campaigns have been performed at room and high temperature. In this work, we focus on shear behaviour to well characterize the modulus evolution as a function of temperature. Then, a numerical analysis is required to precisely identify shear modulus values using ABAQUS code.
In addition, with the aim of determine mechanisms at the mesoscopic scale and to fit numerical material approaches, local strain measuring is still a goal associated with mechanical tests. Non Destructive Technics like X-Ray tomography (Figure 2) and Digital Image Correlation can be achieved; some examples are given for both bending and off-axis compression tests.

Topic: 11 - Thermomechanical behavior and performance of Composites
Abstract no. 1950

# Time-dependent deformation and damage modeling of an orthogonal 3D woven SiC/SiC composite at elevated temperature in vacuum


Presenting author: M IKARASHI Yoshito
Affiliation: Tokyo University of Agriculture and Technology
2-24-16, Naka-cho,184-8588 Koganei-shi (Tokyo, Japan)

Co-authors: OGASAWARA Toshio AOKI Takuy



This study examined the effect of microcrack propagation on the time-dependent deformation behavior of an orthogonal 3-D woven SiC fiber (Tyranno-ZMI)/BN interface/SiC matrix (CVI+PIP-SiC) composite under constant tensile load at elevated temperature in vacuum. The micro-damage evolution and propagation behaviors were evaluated by in-situ observation using an optical microscope at room temperature. Monotonic, load-unload, and constant load tensile tests were conducted at 1200°C in vacuum. The stiffness change under the constant tensile load testing was also measured.
As results, non-linear creep deformation behavior of the SiC/SiC composite was observed under constant tensile load above the proportional limit stress (about 80 MPa). The stiffness gradually decreased with time, which was estimated to be caused by the matrix crack propagation in the transverse (90°) layers.
Microcrack propagation behavior of the SiC/SiC composite at elevated temperature was modeled using shear-lag model for cross-ply and unidirectional composites. A creep deformation model was proposed incorporating the linear-viscoelastic constituent model into the shear-lag model. The predicted creep deformation agreed well with the experimentally obtained results.

Topic: 11 - Thermomechanical behavior and performance of Composites
Abstract no. 2126

# On Performing Digital Image Correlation at Very High Temperatures


Presenting author: Mme BERNY Myriam
Affiliation: LMT, ENS Paris-Saclay, CNRS, Université Paris-Saclay // SAFRAN Ceramics
61 avenue du Président Wilson,94235 Cachan (France)

Co-authors: HERB Vincent (SAFRAN Ceramics), LACOMBE Benjamin (SAFRAN Ceramics), ROUX Stéphane (LMT ENS Paris-Saclay CNRS Université Paris-Saclay), HILD François (LMT ENS Paris-Saclay CNRS Unversité Paris-Saclay)



To increase the performance of future aircraft engines, Safran Ceramics is developing for the hot zones of engines a new material composed of a 3D woven preform and a ceramic matrix. In these applications, ceramic matrix composites (CMCs) are subjected to intense and multiaxial mechanical and thermal stresses that need to be understood and controlled. To this end, multi-instrumented experiments (with cameras) on CMC technological specimens were carried out, during which the material experiences severe 3D thermal loadings (Tmax > 1200°C). In such high-temperature environment, performing Digital Image Correlation (DIC) to measure accurate displacements remains challenging (alteration of speckle pattern, variation of brightness and contrast, increased uncertainties due to heat haze effect). A spatiotemporal approach for DIC was proposed to filter out heat haze effects and correct temporal variations of brightness and contrast.
Global spacetime registrations consist in measuring the spacetime displacement field u(x,t) over an image series (video) that minimise the overall grey level residuals between a reference image and the series of deformed images corrected by the sought displacement field. The displacement field is parameterised as a dyadic product between space and time shape functions. Moreover, the grey level conservation assumption is relaxed in the DIC registration problem through the addition of two corrections (brightness and contrast) fields applied to the reference image.
These procedures were used to measure 3D (2D space + 1D time) displacement fields of CMC samples heated by a flame or a laser beam. Spacetime DIC leads to consistent displacements compared to instantaneous DIC measurements. In addition, the approach removed an important part of convection effects, giving less noisy displacements over time. The real displacements (i.e., unaffected by heat haze) of the sample can consequently be measured with good accuracy and be used for identification purposes with smaller uncertainties on the sought parameters.

Topic: 11 - Thermomechanical behavior and performance of Composites
Abstract no. 2060

# Mechanical Behaviour of Oxide/Oxide Composites and Analysis of Damage by Acoustic Emission


Presenting author: Dr REYNAUD Pascal
Affiliation: INSA Lyon - MATEIS
7 avenue Jean Capelle,bat. Blaise Pascal,69621 Villeurbanne (France)

Co-authors: GUEL Nicolas (Univ Lyon/INSA Lyon/MATEIS Univ Bordeaux/LCTS IRT St-Exupéry), GODIN Nathalie (Univ Lyon / INSA Lyon / MATEIS), CATY Olivier (Univ Bordeaux / LCTS), MAHDI Stéphane (IRT Saint Exupéry), BOUILLON Florent (SAFRAN Ceramics)



Oxide-based composites are interesting materials due to their excellent thermomechanical properties, low density and good resistance to oxidation making them suitable for hot components requirements. But their microstructure is characterised by several kinds of heterogeneities at different scales like: macroporosity, shrinkage cracks, microporosity…, and the aim of this paper is to establish a relationship between the microstructure of these composites and their mechanical behaviour under tensile loading. Hence macroscopic mechanical tests are monitored with acoustic emission to obtain additional informations on the damage mechanisms. Mechanical tests were performed on cross-weave oxide-based CMCs at room temperature in two textile complementary orientations: tests in fiber orientation (0/90°) and ±45° regarding to textile orientation. The AE analysis is based on a signal approach using waveforms from four sensors (2 nano30 and 2 picoHF – MistrasGroup) with complementary frequency ranges. To better understand the link between AE activity and damage mechanisms, in situ tests were also performed with AE monitoring. Hence energetic signals detected during these various tests match with debonding between the plies, and damages at lower scales like matrix cracks and small fiber/matrix debonding were linked to less energetic signals. Signal-based AE analysis coupled with in-situ tests appears therefore to be an interesting approach to better understand the role of the microstructure on the damage behaviour of these CMCs, particularly the effects of delamination and the interactions between yarns inside the plies, and the modifications induced by a change of the plies orientation.

Topic: 11 - Thermomechanical behavior and performance of Composites
Abstract no. 2088

# In-situ observation of contact damage in a SiC/SiC Ceramic Composite

Presenting author: Dr SINGH Shraddha
Affiliation: Postdoc
Department of Materials,University of Oxford,OX1 3PH Oxford (Oxfordshire, United Kingdom)
Co-authors: MARROW James (University of Oxford), PIRZADA Talha (University of Oxford)

Abstract: SiC-SiC ceramic matrix composites are candidate materials for accident tolerant fuel clad in light water nuclear reactors and also for high temperature fuel clad in gas cooled advanced reactors. One key performance requirement is tolerance to local damage from mechanical contact between the ceramic fuel and the clad, which may occur due to irradiation-induced dimensional change of the fuel at high burn-up. Ceramic fibre composites have complex, heterogeneous structures. As a first step towards investigating how local and intense deformations can be accommodated, the progressive development of contact damage has been investigated using in situ high resolution X-ray computed tomography to observe the Hertzian indentation behaviour of a non-irradiated ceramic composite at room temperature. The material was provided by the EU H2020 Matisse project, and is a 45° braided SiC fibre tube with a CVD SiC matrix. The 3D deformation field below the indenter has been quantified by digital volume correlation analysis of tomographs, with the objective of extracting local criteria for matrix, fibre and interface cracking. Such data are required to develop and inform predictive models and test procedures, which will be needed to design and qualify composites for nuclear applications.

Topic: 11 - Thermomechanical behavior and performance of Composites
Abstract no. 1075

# Temperature-dependent fatigue hysteresis behavior of fiber-reinforced ceramic-matrix composites

Presenting author: M LI Longbiao
Affiliation: Nanjing University of Aeronautics and Astronautics
29 Yuda St.,210016 NANJING (China)

Co-authors:

The temperature-dependent fatigue hysteresis behavior of fiber-reinforced ceramic-matrix composites (CMCs) is investigated using the micromechanical approach. The temperature-dependent parameters of the fiber/matrix interface shear stress, fiber and matrix elastic modulus and thermal expansional coefficient, and fiber/matrix interface debonded energy are considered to determine the interface debonding length, interface sliding length and unloading/reloading transition stress. The temperature-dependent fatigue hysteresis loops models are developed, and the relationships between the temperature, fatigue hysteresis loops, interface debonding and sliding are established. The experimental fatigue hysteresis loops of C/SiC, SiC/SiC and SiC/[Si-B-C] composites at different fatigue peak stresses and testing temperatures are predicted using the developed temperature-dependent fatigue hysteresis loops models.

Topic: 11 - Thermomechanical behavior and performance of Composites
Abstract no. 2254

# Inelastic behaviour of ceramic-matrix composites: an Ultrasonic characterisation approach

Presenting author: Prof BASTE Stéphane
Affiliation: I2M, CNRS UMR 5295, Université de Bordeaux
351, cours de la Libération,33405 Talence (France)

Co-authors:

A methodology for formulation of constitutive laws of CMCs is summarised. It relies on an anisotropic damage evaluation that accurately separates the effects of the various mechanisms on the non-linear behaviour. Cyclic loading is subject of a particular attention in order to explain complex form of Hysteresis loops.

The interface slip between fibbers and the matrix existing in matrix cracking, in which matrix cracking and interface debonding occurred in longitudinal yarns, is considered as the major reason for hysteresis loops of CMCs. The extent of the inelastic strains and the area of the hysteresis loops result from both an intense interfacial debonding and fibre-matrix frictional sliding. Limited hysteresis loops account for negligible frictional sliding while debonding, on the other hand, can be broadly present. Thus, interfacial sliding stress and lengths of decohesion are key parameters in the global behaviour.

A micromechanical model used with accurate measurements of the various components of the total strain gives access to the value of the interfacial sliding stress. Experimental variations of the stiffness tensor give access to the intensity of longitudinal damage, i.e., lengths of decohesion. The complex form of hysteresis loops is the result of the opening-closure of the transverse cracks created during loading. By introducing various behaviour thresholds, the constitutive laws of the internal variables chosen describe the three-dimensional anisotropic changes in elasticity as well as the inelastic strains variation under cyclic loading. Various scales can be taken into account. The major point of the methodology lies in the non-arbitrariness of the choice of the internal variables with a concrete physical meaning that reflect the underlying processes on the microscale, as example, the cracks density or the crack opening displacement.

Topic: 11 - Thermomechanical behavior and performance of Composites
Abstract no. 2278

# Damage Evolution in an EBC-SiC/SiC System Due to Laser Heating Induced Thermal Gradients and Mechanical Loading

Presenting author: Mme PIERCE Jennifer
Affiliation: University of Dayton Research Institute
300 College Park,45469 Dayton (Ohio, Ukraine)

Co-authors: ZAWADA Larry (Universal Technology Corporation), PITZ Jeremey (UES), WHITLOW Travis (University of Dayton Research Institute), HUNT Reece (SOCHE), RAPKING Daniel (University of Dayton Research Institute), VERNON Jonathan (Air Force Research Laboratory), JEFFERSON George (Air Force Research Laboratory), PRZYBYLA Craig (Air Force Research Laboratory)

The most promising ceramic matrix composites (CMCs) for efficient propulsion applications are dense composites based on SiC. However, in combustion environments SiC volatilizes and therefore requires environmental barrier coatings (EBCs) for protection. On this program, relationships between the stochastic nature of as-processed microstructures of EBC-CMC systems and the coupled effects of thermal gradients under mechanical load were studied using a unique test capability that can combine fiber laser induced thermal exposure with various loading conditions. The material under test is a SiC/SiC composite fabricated via chemical vapor infiltration (CVI) and densified by melt-infiltration of silicon. The EBC is a dual layer, air-plasma sprayed, coating system based on silicon and ytterbium-disilicate. Both isothermal conditions and rapid thermal cycles were applied using the fiber laser on one face of dogbone test specimens, with and without the EBC, while under uniaxial tensile conditions. In-situ damage monitoring technologies that included radiometry, digital image correlation, and acoustic emission detection were implemented during testing. Matrix cracking damage due to coupled thermo-mechanical exposures and microstructural features were studied using registered data from the different sensing techniques. Post-test microstructural studies documented crack size, spacing, and location for the different thermos-mechanical conditions. Single-sided laser heating generates a complex state of stress through the thickness and along the length, and results in damage evolution that is distributed different from that observed during isothermal testing. Thermo-mechanical simulations were compared to the material response for the EBC-SiC/SiC and uncoated SiC/SiC systems.

Topic: 11 - Thermomechanical behavior and performance of Composites
Abstract no. 2010

# Low temperature mechanical properties and oxidation behavior of melt-infiltrated SiC/SiC ceramic matrix composites

Presenting author: Mme DETWILER Kaitlin
Affiliation: University of Virginia
395 McCormick Road,22904 Charlottesville (VA, United States)
Co-authors: OPILA Elizabeth (University of Virginia)

SiC/SiC ceramic matrix composite (CMC) turbine engine components will experience a wide range of temperatures, environments and stresses due to thermal gradients and flight operating conditions. Historically, studies on SiC/SiC CMC oxidation and mechanical properties have been conducted near their upper use temperature, around 1200 C. The present work is focused on a lower temperature, higher stress regime where water vapor is present. It is vital to understand the coupling effects of applied stress and oxidation at lower temperatures, but first decoupled baseline behavior needs to be understood. Fast fracture tensile tests were conducted to specimen failure in lab air to measure baseline mechanical properties at relatively low temperatures of 400, 600, and 800 C. Acoustic emission was utilized during tensile testing to track the onset of matrix cracking within the composite. Oxidation kinetics were separately evaluated using thermogravimetric analysis (TGA) to track weight change of specimens with exposed fibers on machined edges during low temperature exposures at the same temperatures in dry and wet oxygen. Resulting microstructures for both the tensile and TGA specimens were characterized via scanning electron microscopy. Mechanisms for oxidation and mechanical behavior will be explored providing guidance for future coupled oxidation load hold experiments.

Topic: 11 - Thermomechanical behavior and performance of Composites
Abstract no. 1832

# Fatigue behavior and damage analysis of a LSI based 2.5D C/C-SiC composites

Presenting author: Dr LI Yang
Affiliation: Powder Metallurgy Research Institute, Central South University
Lushan south road 932,410083 Changsha (Hunan, China)

Co-authors: XIAO Peng (Powder Metallurgy Research Institute Central South University), ALMEIDA Renato (Advanced ceramics University of Bremen), ZHOU Wei (Lehrstuhl Keramische Werkstoffe Universität Bayreuth), LANGHOF Nico (Lehrstuhl Keramische Werkstoffe Universität Bayreuth), LI Zhuan (Powder Metallurgy Research Institute Central South University)

As the essential requirement of significant weight-reduction in Chinese future high speed train with speed higher than 400 km/h, liquid silicon infiltration(LSI) based C/C-SiC composites shows great potential as brake materials because of the advantages of light-weight, excellent thermal resistance, favorable mechanical strength and toughness, stable frictional resistance and relatively low cost. Before practical running operations, investigation regarding to fatigue performance and damage analysis is urgently in need and also of great significance.

In this work, investigation regarding fatigue behavior including S-N curve, hysteresis loops, residual strength and mechanical response of post-fatigue specimen, microstructural characteristics and damage analysis via A.E. monitoring, etc., for a classic chemical vapor infiltration and LSI based 2.5 dimensional reinforced C/C-SiC (2.5D C/C-SiC) composites will be presented in details.

Topic: 11 - Thermomechanical behavior and performance of Composites
Abstract no. 2079

# The research of 2D-C/SiC&2D-SiC/SiC tensile properties under the combined environment of mechanical stress, high temperature, oxygen atmosphere.

Presenting author: M PI Huilong
Affiliation: Beijing aerospace technology institute
Yungang street,Feitai district,100074 beijing (Beijing, China)

Co-authors:

Ceramic matrix composites ?CMCs?have high temperature capability, high strength at elevated temperatures, high toughness, light weight, and environment durability. Therefore?CMCs also was considered to be one kind of the most important material candidates for the hot-structure. Take consider that CMCs always were used under the combined environment of force, high temperature, oxidation and moisture,

It is necessary to study the properties of CMCs under the combined environment of mechanical stress, high temperature, oxygen atmosphere. In this paper, 2D-C/SiC and 2D-SiC/SiC high temperature tensile properties in the air were tested?evolution of material microstructure and tensile properties were researched. Research results show that CMCs high temperature durability is controlled by the matrix cracking stress, the temperature oxidation resistance of the fiber. Research results show that 2D-C/SiC was fractured within 3 hours under 80MPa/700-1000?. The fracture time of the 2D-SiC/SiC was greater than 100 hours under 100MPa/500-1000?.

Topic: 11 - Thermomechanical behavior and performance of Composites
Abstract no. 2016

# Thermomechanical characterization of 3D needdle Carbon/Carbon composites at very high temperature for space application.


Presenting author: Mme BILLARD Capucine
Affiliation: LCTS
3 allée de la Boétie,33600 Bordeaux (France, France)

Co-authors: CATY Olivier (LCTS), MARTIN Eric (LCTS)



Ceramic Matrix Composites (CMC) are high performance materials displaying remarkable properties such as low density and excellent thermomechanical behavior at very high temperatures (2000°C). They have recieived increasing attention in the past decades especially for aerospace application or airplane braking systems.
The investigated materials are three-dimensional needdle Carbon/Carbon composites (3D C/C) used for propulsion nozzle throat of rockets developed by ArianeGroup. The main objectives of this research are to understand the thermomechanical and viscoelastic properties and investigate the failure events at high temperature in order to develop a predictive model.
Previous studies have examined the mechanical behaviour of this type of materials, but the microstructural characteristics and mechanical properties of 3D C/C have been less studied than other CMCs. The detailed understanding of mechanical behavior, its link with the microstructure of the material until 2000°C lack for a complete study. To understand and predict the behavior of the material in use, precise observations of the microstructure as well as on a good understanding of damage mechanisms at different scales have to be performed.
To achieve this goal, in-situ tensile tests under X-ray synchrotron micro-tomography (?CT) have been carried-out on specimens from 25°C up to 2000°C with a specific device. Coupling X-ray ?CT and mechanical tests is a powerful technique. Synchrotron radiation appears as a necessity to obtain high resolution which provides rich 3D information. Damage and failure events are quantified and related to the architecture of the 3D C/C to propose scenario. Digital volume correlation (DVC) is used for analyzing cracks by studying the discontinuities of the mechanical fields. In addition, MEB and Push-out characterizations will be performed to analyze surfaces and interfacial behavior.
Results of this in-situ tensile tests

Topic: 11 - Thermomechanical behavior and performance of Composites
Abstract no. 2695

# Building the silicon carbide nanowire network on the surface of carbon fibers: enhanced interfacial adhesion and high-performance wear resistance


Presenting author: Prof FU Yewei
Affiliation: Northwestern Polytechnical University
No.127 West Youyi Road,710072 Xi'an (China)
Co-authors: LE Zhou (Northwestern Polytechnical University xi'an 710072 China), TAO Yin (Northwestern Polytechnical University xi'an 710072 China)



In order to improve the tribological properties of carbon fiber (CF) reinforced resin matrix composites (CFRRMC), in-situ grown silicon carbide nanowires (SiCnws) were established on the surface of CF. The contact angle of the modified CF with the resin was reduced by 32° compared to the original. The grafted SiCnws could bridge the CF and resin and form a network on the surface of CFRRMC, greatly improving their interfacial bonding and effectively transferring loads. More importantly, the SiCnws-CFRRMC has the better stability of coefficient of friction. Further, the wear rate of SiCnws-CFRRMC was significantly reduced by 76%, which is attributed to the friction film and reduced fatigue wear. This work enriches the approach to modification for surface of CF and provides an effective way to obtain high-performance materials by building networks, extending the applications of CFRRMC

Topic: 11 - Thermomechanical behavior and performance of Composites
Abstract no. 1876

# High-temperature oxidation behavior and mechanism of C/SiBCN composites in static air


Presenting author: Dr CHEN Si'an
Affiliation: Science and Technology on Advanced Ceramic Fibers and Composites Laboratory, National University of Defense Technology
Deya Road 109,410073 Changsha (China)

Co-authors: JIA Yan GOU Yanzi HU Haifen



C/SiBCN composites are increasingly concerned in high-temperature applications for excellent oxidation resistance of precursor-derived SiBCN ceramics up to 1600 °C. C/SiBCN composites prepared by precursor infiltration and pyrolysis were oxidized in static air at 1200-1700 °C. The oxidation microstrusture and behaviors were investigated, and two different oxidation mechanisms for the low (1200-1500 °C) and high (1600-1700 °C) temperature section were raised. At 1200-1500 °C, the fibers were preferentially oxidized. The weight loss rate of the composites was highest (54.6%) after oxidation at 1500 °C for 60 min, due to the completely oxidation of carbon fibers. An oxide scale with growth rate constants of 5-7 ?m2/h appeared around the channels (formed by the oxidation of fibers) after oxidation at 1500 °C for 60 min. At 1600-1700 °C, a SiO2 oxide layer formed due to the rapid oxidation of the matrix. The oxidation rate of the composites at 1600-1700 °C was controlled by the diffusion rate of oxygen through the SiO2 oxide layer. The weight loss rate of composites decreased to 28.6% after oxidation at 1600 °C for 60 min, due to the formation of a dense SiO2 oxide scale.

Topic: 11 - Thermomechanical behavior and performance of Composites
Abstract no. 2632

# Curved Beam Strength of Silicon Melt Infiltrated SiC/SiC Composites


Presenting author: M SCHMIDT Jens
Affiliation: Fraunhofer ISC
Gottlieb-Keim-Straße 62,95448 Bayreuth (Germany)

Co-authors: KONSCHAK Alexander (Fraunhofer ISC), BERNSTEIN Wolfgang (Fraunhofer ISC), SCHMIDT Manuel (Fraunhofer ISC), SCHMIDT Jens (Fraunhofer ISC)



New CMC materials are the key for the development of the next generation of highly efficient aero-engines. Therefore, different types of SiC/SiC ceramics are currently under development. High strength, high stiffness as well as high strain to failure are required. To guarantee the safety and reliability of such CMC parts, the fracture behavior is one of the key-issues which have to be clearly understood. The ASTM standard D6415 can be used to study the correlation between the fibre lay-up and fracture behavior of curved beam samples. The curved beam consists of two straight legs connected by a 90° bend with a 6.4 mm radius. For the mechanical testing, a Hegewald and Peschke universal testing machine Inspekt Table 100 with a special setup according to the standard was used. A complex stress state with an out of plane tensile stress component is produced in the curved area of the specimen when force is applied. Precise bearing fixtures with a stationary head and a movable head are essentially to perform the test. For our research work, curved beam specimens were manufactured by axial warm pressing of prepregs and a silicon melt infiltration (MI) process. Both composites based on unidirectional layers and 2D fabrics were tested. Samples with dimensions of 90x25x2 $mm^3$ were prepared and tested both after curing and after MI in their ceramic stage. Different stacking sequences in the thickness of the composites were adjusted. Symmetrical lay-up was obtained by the combination of 0°, 45° and 90° unidirectional layers. A correlation between the fiber lay-up and the bending strength could be concluded from the force-displacement curves. The microstructure and failure plane of the broken parts were investigated by using SEM. Delaminations were detected by 3 D computed tomography.

# Topic: 12

# Nondestructive Testing and Health Monitoring of Ceramic Composites

Topic: 12 - Nondestructive Testing and Health Monitoring of Ceramic Composites
Abstract no. 2290

# The Effects of Cooling Holes on SiC/SiC Strength and Durability

Presenting author: Dr SMITH Craig
Affiliation: NASA Glenn Research Center
21000 Brookpark Rd.,MS 106-5,44135 Cleveland (OH, United States)

Co-authors: BHATT Ramakrishna (Ohio Aerospace Institute), KALLURI Sreeramesh (Ohio Aerospace Institute), PRESBY Michael (NASA Glenn Research Center)

Ceramic Matrix Composites (CMCs) such as SiC/SiC are currently operating in select high temperature components of turbine engines. When these materials are implemented in components at higher operating temperatures, film cooling may be necessary. Film cooling requires holes to be fabricated at appropriate locations within these components. This study examines the effect of potential cooling holes on SiC/SiC strength and durability. Mechanical test data on specimens with hole(s) is reported for isothermal conditions at room and elevated temperatures. Several hole geometries fabricated in SiC/SiC samples are explored. Tools such as Digital Image Correlation (DIC) and Acoustic Emission (AE) are used to monitor strain and cracking upon loading.

Topic: 12 - Nondestructive Testing and Health Monitoring of Ceramic Composites
Abstract no. 2098

# Correlating damage to the microstructure of SiC/SiC CMCs using x-ray tomography techniques

Presenting author: Mme HILMAS Ashley
Affiliation: University of Michigan
4943 Lakeridge St.,Apt 2B,48197 Ypsilanti (MICHIGAN, United States)

Co-authors: SEVENER Kathleen (University of Michigan), HALLORAN John (University of Michigan)

Due to their high temperature capabilities, high toughness, and their light weight, SiC/SiC CMCs are currently being used in aerospace applications. X-ray tomography techniques have recently been utilized as a method to study these composite materials in order to better understand their mechanical properties. In this study, MI SiC/SiC CMCs were characterized through the use of synchrotron x-ray tomography at the Advanced Light Source (ALS) at Lawrence Berkeley National Laboratory and lab scale x-ray tomography using a Zeiss Xradia. These techniques allowed us to correlate damage progression to various microstructural features in the SiC/SiC CMC unidirectional specimens including.

Topic: 12 - Nondestructive Testing and Health Monitoring of Ceramic Composites
Abstract no. 2118

# Ultrasonic NDT and SHM methods for composites at high temperature using optical fiber sensors


Presenting author: Prof OKABE Yoji
Affiliation: Institute of Industrial Science, The University of Tokyo
IIS Dw-401,4-6-1 Komaba, Meguro-ku,153-8505 Tokyo (Japan)
Co-authors: YU Feng-ming (Institute of Industrial Science The University of Tokyo), SAITO Osamu (Institute of Industrial Science The University of Tokyo)



The development of reliable heat-resistant composite materials requires the use of nondestructive testing (NDT) techniques for evaluating the damage progress during material testing at elevated temperatures. Furthermore, structural health monitoring (SHM) technologies that can operate at high temperature are expected to be realized for monitoring heat-resistant composite structures.
In recent years, the authors have developed an optical fiber ultrasonic sensing system with high sensitivity and broadband response for strain waves based on a phase-shifted fiber Bragg grating (PSFBG). Since glass optical fibers have high heat resistance, this system was improved for remote sensing of acoustic emission (AE) signals at high temperature by using the optical fiber as an ultrasonic waveguide from an object material in a high-temperature furnace to the PSFBG placed far from the furnace. As a result, AE signals at about 1000 degrees Celsius were successfully detected keeping their precise waveform, because the AE waves propagated in the optical fiber as a pure longitudinal mode. Therefore this method will be useful for NDT and SHM at high temperature to evaluate the damage progress in heat-resistant materials.
We are also investigating the application of a laser ultrasonic visualizing inspector (LUVI) developed by Tsukuba Technology to NDT at high temperature. The LUVI can visualize the propagation behavior of ultrasonic guided waves in complex shape structures. In order to enhance the heat resistance of the system, the piezo-ceramic sensor in the LUVI was replaced by our optical fiber ultrasonic sensor with the remote sensing configuration. As a result, the improved LUVI was able to visualize the ultrasonic wave propagation in a metal plate at 350 degrees Celsius. Presently, we are attempting to observe the wave propagation at about 1000 degrees Celsius. This system has a potential to be a promising NDT method for composites in a high-temperature environment.

Topic: 12 - Nondestructive Testing and Health Monitoring of Ceramic Composites
Abstract no. 1842

# Prognostic and Health Management (PHM) for ceramic matrix composite with acoustic emission during long-term mechanical tests at intermediate temperature


Presenting author: Dr GODIN Nathalie

Affiliation: INSA of Lyon, MATEIS
7 avenue Jean capelle,69621 Villeurbanne cedex (France)

Co-authors: REYNAUD Pascal (INSA of Lyon MATEIS), FANTOZZI Gilbert (INSA of Lyon MATEIS)



Damage of composite materials is a key factor for the durability in service. It is therefore essential to define suitable damage indicators and to develop models to estimate the Remaining Useful Lifetime (RUL) from analysis of precursor events resulting from damage. Acoustic emission is relevant to the development of the PHM because it allows knowing the state of damage of a composite structure in real time. This approach is based on two phases: diagnostic and prognostic. This work is dedicated to lifetime prediction using AE for long-term tests on CMC during static and cyclic fatigue tests at high temperatures. New indicators of damage have been defined, based mainly on acoustic energy analyses. These indicators highlight critical times (around 20 % for the cyclic fatigue tests and 50 % of the composite lifetime for the static fatigue tests) allowing an evaluation of the remaining lifetime. Moreover, the clustering of acoustic emission, using a supervised clustering method based on random forest approach, makes possible to get a real-time detection of each damage phenomena and to identify the mechanism responsible for this critical time. This analysis pointed out different damage mechanisms generated by cyclic loading, which are mainly debonding and friction at matrix/fibre and matrix/matrix interfaces. Furthermore, a link is established between characteristic time at 20 % for the cyclic fatigue and the beginning of the matrix cracking. For the static fatigue test, the critical time around 50 % corresponds to the delayed failure of thermally aged fibres (slow crack growth, oxidation). To perform RUL prognostics, the results obtained with these new indicators can be used to propose new predictive laws of lifetimes complementary to the classical Benioff's law. Nevertheless, the determination of the acoustic signatures and characteristic times are linked to testing conditions and specimen geometries, requiring investigations by modelling, as done already on the effects of specimen's sizes.

Topic: 12 - Nondestructive Testing and Health Monitoring of Ceramic Composites
Abstract no. 2208

# New X-ray Imaging system using a Talbot-Lau Interferometer for damage detection of CMCs


Presenting author: Dr KOTANI Masaki

Affiliation: Japan Aerospace Exploration Agency
6-13-1,Osawa,181-0015 Mitaka (Tokyo, Japan)

Co-authors: SATO Mitsuhiko (The Center for Ceramic Matrix Composites (CCMC), Tokyo University of Technology), TANAKA Yoshihisa (The Center for Ceramic Matrix Composites (CCMC), Tokyo University of Technology), FIJIWARA Chikara (The Center for Ceramic Matrix Composites (CCMC), Tokyo University of Technology), HATTA Hiroshi (The Center for Ceramic Matrix Composites (CCMC), Tokyo University of Technology), SHICHIJO Naohiro (The Center for Ceramic Matrix Composites (CCMC), Tokyo University of Technology), KAGAWA Yutaka (The Center for Ceramic Matrix Composites (CCMC), Tokyo University of Technology)



An X-ray imaging system based on Talbot-Lau interferometry has been developed and its feasibility for direct monitoring of damage evolution process in CMCs (SiC/SiC, C/SiC, OX/OX etc.) has been studied. Obtained X-ray images, such as differential phase contrast images and scattered images etc., are processed using advanced image analysis systems. The results are compared with a conventional X-ray image system so that the developed Talbot-Lau interferometer device is useful for detection of matrix cracks, delamination, and pores, especially under low magnification. CMCs are subjected to tensile loading and the system is also applied for direct detection of damage evolution behavior of CMCs. A set of obtained images clearly demonstrated typical damage evolution behavior of CMCs.

# X-ray microtomography characterization of damage and crack healing mechanisms in SiCf/Si-B-Cm composites during static fatigue

Presenting author: M EBEL André
Affiliation: LCTS
3, Allée de la Boétie,33600 Pessac (France)
Co-authors: CATY Olivier (Laboratoire des composites thermostructuraux (LCTS),
REBILLAT Francis (Laboratoire des composites thermostructuraux (LCTS)

The crack healing capability of composites with a self-healing Si-B-C matrix, Nicalon fibers and pyrocarbon (PyC) interphase was studied by means of static fatigue experiments, electrical resistance (ER) monitoring and X-Ray micro-computed tomography (µCT). Two sets of experiments were performed: static fatigue tests with periodic unloading-reloading cycles followed with post-mortem µCT scanning and in-situ µCT static fatigue tests. ER monitoring was implemented on both type of experiments. Mechanical tests were performed under a constant 100 MPa load at 650 and 800°C in ambient and humid air (10 vol.% H2O). For each kind of experiment, mechanical load was applied after heating and unloading was only performed after cooling at the end of the test in order to solidify the glassy sealant before crack closure. Observations of the composite cross-section before and after testing were performed by means of optical microscopy, SEM, EDS and Raman spectroscopy.
Post-mortem µCT scanning on interrupted experiments were used to localize cracks and observe the sealant distribution within the cracks. In-situ µCT static fatigue test were used to scan the sample at different load levels during loading in order to highlight the damage mechanism. Scans at different times for a constant load were also used to follow the crack healing according to time. Crack healing observed on µCT scans could be correlated with ER variation (?R) and modulus variation (?E). In ambient air, fast healing leads to low ?R and ?E. In humid air, volatilization of the sealant prevents complete healing, leading to large ?R and ?E. However, crack healing in humid air was more pronounced at 800°C than at 650°C, leading to lower ?R and ?E. This phenomenon has been correlated with the higher silica content within the sealant at 800°C which limits its volatilization.

Topic: 12 - Nondestructive Testing and Health Monitoring of Ceramic Composites
Abstract no. 1973

# Deep Learning CT Image Segmentation for High Throughput Quantitative Analysis of SiC-SiC Ceramic Matrix Composites


Presenting author: M BADRAN Aly
Affiliation: University of Colorado Boulder
UCB 426 Room 30,80309 Boulder (Colorado, United States)

Co-authors: MARSHALL David (University of Colorado Boulder), MARSH Mike (Object Research Systems), LEGAULT Zacharie (Polytechnique Montreal), MAKOVETSKY Ruslana (Object Research Systems), PROVENCHER Benjamin (Object Research Systems), PICHE Nicolas (Object Research Systems)



Computed Tomography (CT) imaging has become a useful tool for characterizing the internal microstructure and the nature of damage in Ceramic Matrix Composites (CMCs), because of its capability to non-destructively provide 3D images with sub-micron resolution. In recent years, progress has been made in extracting quantitative details from such images by segmenting the different phases of the CMC using image processing and reconstruction techniques. However, past approaches have difficulties when the material phases of the composite show very weak or no contrast, as is the case in SiC-SiC composites. In this work, we present a new Deep Learning (DL) approach for efficiently segmenting CT images of CMCs. With the DL solution, users train a neural network model with a few manually highlighted cross-sections of a CMC, and then the model accurately labels the material phases on the remainder of the thousands of slices in a stack of stitched CT images. To make our DL system accessible by those with no DL expertise, we integrate our solution into a widely-used user-friendly visualization and image processing platform. We used seventeen manually curated slices to train an FCNDenseNet convolutional neural network model for no more than 200 training iterations, and achieved greater than 99% labeling accuracy; the trained model is capable of labeling previously unseen images at a rate of 29 per minute on a standard workstation GPU. The outcome of these segmentations is used to provide rich quantitative information, including reports of volume fractions of different phases and pore size distributions. We show 2D and 3D renderings of the DL segmentation, executed a merged stack of greater than 5000 slices, to demonstrate the efficacy of this approach. We share our trained DL model in a public repository so other workers can apply the same model to their own CMC image data.

Topic: 12 - Nondestructive Testing and Health Monitoring of Ceramic Composites
Abstract no. 1941

# Unsupervised acoustic emission data clustering during creep tests of 2D-C/SiC in a wet oxidizing atmosphere


Presenting author: Prof YAO Leijiang
Affiliation: Northwestern Polytechnical University
127 Youxi West Road,710072 Xi'an (Shaanxi, China)

Co-authors: LYU Peng (Northwestern Polytechnical University), BAI Guodong (Northwestern Polytechnical University), LI Bin (Northwestern Polytechnical University), AUGOUSTI Andy (Kingston University)



The failure process of carbon reinforced silicon carbide (C/SiC) composites in elevated temperature involves complex multiple microscopic fracture mechanisms. The use of Acoustic Emission (AE), being almost the only technique to realize on-line damage monitoring, provides an effective tool for identifying these damage modes and describe their evolution. Stress rupture tests of 2D-C/SiC were tested under conditions of 1300?C and a wet oxygen atmosphere. AE data was collected during the whole loading process. To discriminate the physical mechanism of each AE event, principal component analysis (PCA) and fuzzy clustering algorithm were used to classify all the AE events into several groups. Each group was then identified with a particular damage mode according to its AE features and an empirical understanding of the damage behavior of C/SiC. The test results show that creep life has significant variations for different samples under the same test conditions. AE events were divided into four clusters, distinguished on the basis of the difference in their AE characteristic parameters. The four clusters correspond to matrix cracking, interfacial damage, fiber breakage and fiber bundle breakage respectively. Generally, the accumulation of AE events experiences three stages, namely an initial rapid increase, a period of relative stability and then a final increase. However, for samples with different lifetimes, the specific evolution of acoustic emission is different in its detail. It is found that the percentage of AE events corresponding to fiber breakage has an inverse relationship with rupture lifetime. The reason is hypothesized to be due to the extension and distribution of oxidation within C/SiC under different creep lifetime conditions.

Topic: 12 - Nondestructive Testing and Health Monitoring of Ceramic Composites
Abstract no. 2220

# Understanding Damage Mechanisms of CMC Blade Root Sub-elements


Presenting author: Dr JONES Eric
Affiliation: Air Force Research Laboratory
2941 Hobson Way,45433 Wright-Patterson AFB (OH, United States)

Co-authors: JEFFERSON George (Air Force Research Laboratory), PIERCE Jennifer (University of Dayton Research Institute), ZAWADA Larry (Universal Technology Coorperation), WHITLOW Travis (University of Dayton Research Institute), PRZYBYLA Craig (Air Force Research Laboratory)


Understanding Damage Mechanisms of CMC Blade Root Sub-elements


Eric Jones1, George Jefferson1, Jennifer Pierce2, Larry Zawada3, Travis Whitlow2 and Craig Przybyla1
1Air Force Research Laboratory, Materials and Manufacturing Directorate, WPAFB, OH
2University of Dayton Research Institute
3Universal Technology Corporation



While much of the current research on ceramic matrix composites (CMC) uses flat panels for experiments, CMC components are often highly complex shapes in advanced turbine engine applications. Components such as turbine blades and vanes may require ply drops, curved plies, and/or matrix rich regions which have not been studied extensively. Additionally, the complexity of the CMCs architecture could cause other manufacturing defects such as porosity and ply wrinkles that can affect the components durability. The objective of this work is to study the damage evolution of a CMC blade root sub-element under monotonic and fatigue loading at room temperature. The SiC/SiC sub-element investigated has a generic turbine blade root geometry with a complex stress state similar to those of actual turbine blade attachments due to ply drops, matrix rich regions, and porosity. In situ diagnostic tools such as digital image correlation (DIC) and acoustic emission (AE) were used to capture damage initiation and progression. The results show how significantly the sub-element laminate architecture influences damage evolution.

Topic: 12 - Nondestructive Testing and Health Monitoring of Ceramic Composites
Abstract no. 2022

# Real-time non-destructive damage evaluation of C/SiC composites during fatigue loading

Presenting author: Dr LI Bin
Affiliation: National Key Laboratory of Science and Technology on UAV, Northwestern Polytechnical University
127 Youyi West Road,710072 Xi'an (Shaanxi, China)

Co-authors: YAO Leijiang (National Key Laboratory of Science and Technology on UAV Northwestern Polytechnical University), TONG Xiaoyan (National Key Laboratory of Science and Technology on UAV Northwestern Polytechnical University)

C/SiC composites have advantages such as high specific strength/stiffness at high temperature for thermal structure applications, and damage evaluation is needed. With the development of non-destructive testing, real-time non-destructive technologies were used for damage evaluation. 2D plain woven C/SiC composites were performed and monitored by infrared (IR) camera and acoustic emission (AE) during fatigue tests in this paper. In the room temperature, Plain plate and center-holed specimens were performed on Instron8801 fatigue machine at stress ratio of 0.1. IR thermography was recorded using an infrared camera (FLIR ThermaCAM SC3000) with 320×240 pixels at 50 frames per second. AE signal was detected by two acoustic emission wide band sensors, and an AE 2ch. DAQ system (PAC PCI-2 system) was used to record and process the AE data. The infrared camera and AE 2ch. DAQ system were triggered with the +12V to synchronously record the data when the test started. Damage evolution was discussed based on the modulus, AE hits and energy and thermal dissipation Q. Thermal dissipation was closely related to microstructural damage of composites. At low stress level, Q rose in the first cycles and then the rate of Q accumulation gradually approached steady. When the stress level exceeded the limit of fatigue strength, Q rose quickly until the composites failed. Higher applied stress would cause more damage in the the composites, and more AE hits and energy were detected. Compared with modulus, Q and AE energy had fairly well agreement with the damage evolution. It is possible to employ these non-destructive evaluation methods as in-situ damage evolution indicators for 2D C/SiC composites.

Topic: 12 - Nondestructive Testing and Health Monitoring of Ceramic Composites
Abstract no. 1899

# Research progress on non-destructive testing of ceramic matrix composites

Presenting author: Prof MEI Hui
Affiliation: Northwestern Polytechnical University
No 127 Youyi Westroad,710072 Xi'an (Shaanxi, China)

Co-authors:

Ceramic matrix composites (CMC) have been widely used in aerospace, nuclear energy, brake discs and other fields due to their low density, high-temperature resistance, high specific strength and oxidation resistance. CMC has the characteristics of heterogeneous and various structures, complicated fabrication process and harsh service environment, which inevitably results in various defects in materials and components. CMC defects mainly include material defects, structural defects, and environmental damage defects. Various types of defects have different effects on the mechanical properties and safety of materials. Through the non-destructive testing of CMC components such as turbine blades, core cladding tubes, aircraft brake discs, riveted structures, engine tail nozzles and transmission shaft, the defect characteristics of axisymmetric bodies, planes, complex surfaces and other structures are investigated. The typical defects like delamination defects, density defects and inclusion defects are prefabricated in the material, and the influence of materials and structural defects on the mechanical properties of CMC is discussed. Besides, the effects of environmental damage defects on the mechanical and antioxidation properties of CMC were studied through environmental assessment such as oxidation and high temperature.

Topic: 12 - Nondestructive Testing and Health Monitoring of Ceramic Composites
Abstract no. 1843

# Investigation of degradation behavior of ceramic matrix composites by acoustic emission analysis

Presenting author: Dr HAUG Simon
Affiliation: MTU Aero Engines
Dachauer Str. 665,80995 München (Bavaria, Germany)

Co-authors: REICHERT Florian (Schunk Kohlenstofftechnik GmbH), ALBERT Benedikt (MTU Aero Engines), SIKORSKI Siegfried (MTU Aero Engines)

Ceramic matrix composites (CMC) are a promising material class for high tempera-ture application in aerospace industry. Degradation behavior and oxidation re-sistance play a crucial role for the lifetime expectancy of such composites and have to be carefully evaluated.
In this study we investigate the degradation behavior of CMCs with acoustic emis-sion analysis. To this end, tensile test were performed at room temperature prior to and post high temperature exposure to an oxidative environment. The tensile test were accompanied by acoustic emission analysis and recorded signals were classi-fied by a multi-feature pattern recognition algorithm to better understand the mecha-nisms governing failure behavior.

Topic: 12 - Nondestructive Testing and Health Monitoring of Ceramic Composites
Abstract no. 1943

# Characterization of Damage Evolution in CMCs with Acoustic Emission in-SEM


Presenting author: Mme SWAMINATHAN Bhavana
Affiliation: University of California, Santa Barbara
6520 El Colegio Rd,Apt 2226,93117 Goleta (California (CA), United States)

Co-authors: ALMANSOUR Amjad (NASA Glenn Research Center), KISER James (NASA Glenn Research Center), SEVENER Kathleen (University of Michigan), DALY Samantha (University of California Santa Barbara)



Silicon carbide / silicon carbide ceramic matrix composites (SiC/SiC CMCs) are advanced ceramics that are well-suited for the extreme environment conditions of space and aerospace applications due to their low weight, creep resistance, damage tolerance, and high specific strength. In CMCs, the initiation and accumulation of damage depends on characteristics of the constituent landscape including porosity, constituent properties, geometric and structural variations, and microstructural interactions. In order to accurately life CMCs, it is critical to understand the evolution of damage and to characterize which early damage phenomena subsequently lead to crack coalescence and macroscopic failure. In this research, SiC/SiC minicomposites were characterized through a novel experimental approach combining acoustic emission (AE) with tensile testing in-SEM (scanning electron microscope), in order to examine early damage initiation (i.e. below the proportional limit) and its evolution. Damage was characterized at the surface and subsurface, both in the specimen bulk and at the microscale. Surface damage was documented within the gage at incremental stresses via high resolution SEM imaging. AE activity (sound waveforms generated by CMC damage events) were recorded throughout the loading profile. These events were analyzed to determine their axial location along the minicomposite and magnitude, enabling correlation between SEM-observed damage with AE, and an assessment of the relationships between AE spectrum signature characteristics and damage mechanisms such as matrix cracking, fiber bridging, and fiber breakage. This multi-modal approach significantly improved the spatial correlation between an AE event and its associated damage, and facilitated examination of the impact of the constituent landscape on the evolution of damage to final failure. Micromechanical models from the literature were reviewed to assess the applicability to modeling of the observed matrix cracking behavior, including the measured stress-dependent crack opening displacement, and crack spacing and density.


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Topic: 12 - Nondestructive Testing and Health Monitoring of Ceramic Composites
Abstract no. 1822

# Visualisation and Assessment of Matrix Crack Accumulation in a SiCf/SiC CMC via In-situ X-ray Computed Tomography


Presenting author: Dr QUINEY Zak
Affiliation: Swansea University Institute of Structural Materials
029 ISM, Swansea University,Bay Campus, Fabian Way,SA1 8EN Swansea (United Kingdom)
Co-authors: WESTON E. (TWI Technology Centre Wales), NICHOLSON P. I. (TWI Technology Centre Wales), PATTISON S. J. (Rolls Royce plc), BACHE M. R. (Swansea University Institute of Structural Materials)



In order to enhance the understanding of transverse matrix cracking and damage progression on the macroscopic scale, X-ray computed tomography (XCT) imaging and analysis have been performed in conjunction with a commercially available in-situ mechanical loading device on a 0/90° fibre reinforced SiCf/SiC CMC. Test coupons were subjected to a stepped monotonic tensile load that provided accumulating matrix cracking damage, but did not induce rupture. Attempts to measure and quantify the resulting damage using volumetric image analysis techniques are presented, resulting in a correlation of the crack opening displacement where XCT images have been acquired at both the maximum and minimum condition during a selected load cycle. Furthermore, an assessment of the extent to which matrix crack saturation has been reached under the present conditions is discussed. The results are seen as an important step towards correlating the damage behaviour detected via different NDE and health monitoring techniques, for example acoustic emission.

Topic: 12 - Nondestructive Testing and Health Monitoring of Ceramic Composites
Abstract no. 2228

# Measurement of the electrical resistivity to monitor the oxidation propagation inside damaged SiC/SiC composites during ageing under oxidizing environments


Presenting author: M REBILLAT Francis
Affiliation: University of Bordeaux, LCTS
3 rue de la boetie,33600 Pessac (France)

Co-authors: SIMON Coraline (LCTS), CAMUS Gérald (LCTS)



The introduction of Ceramic Matrix Composites parts in civil aeronautics requires a thorough understanding of their damage evolution under the oxidizing environments present within the engines. The development of non destructive evaluation techniques such as acoustic emission or electrical resistivity is therefore essential.
In this respect, the electrical resistivity of a SiC/PyC/[Si-B-C] specimen was monitored during a room temperature tensile test; a good correlation was found between the real time resistance value and the damage state of the material (crack density and debonding density). Electrical resistivity monitoring could then be performed during ageing tests of a few hundreds of hours, under various environments (450°C, ambient and moist air at 10 kPa of water pressure) and mechanical conditions (maximum stress of 100 MPa with creep testing or cyclic fatigue). It appeared that the oxidation of an essential constituent of the composite, the pyrocarbon interphase, led as well to an increase of the electrical resistivity of the specimen. Electrical resistivity monitoring is hence a promising technique allowing a real time estimation of the oxidation of the interphase and the damage state of the material.

Topic: 12 - Nondestructive Testing and Health Monitoring of Ceramic Composites
Abstract no. 2262

# Acoustic Emission Methodologies for Damage Identification and Location in CMCs


Presenting author: Prof MORSCHER Gregory
Affiliation: University of Akron
Mechanical Engineering Department,44325 Akron (OH, United States)
Co-authors: RAZAVI Seyed (University of Akron)



Acoustic emission has been established as a very effective technique as to the identification of when damage such as matrix cracking first occurs in materials, especially ceramic matrix composites. In order to fully utilize the information that is generated by the stress-waves produced by different damage mechanisms (e.g., transverse matrix cracks, shear cracks, fiber breaks, etc..) improved techniques are required to discern waveform features which can be used to identify sources. In addition an appreciation of the material effects (e.g., modulus/density, geometry, fixed points…) must be taken into account as well as stress history. Finally, exact location of sources requires effective time-of-arrival determination which can be very difficult due to various waveform noise sources and the need to process thousands of events. Some advancements and improvements in all of these areas will be discussed as they pertain to CMC testing and analysis.

Topic: 12 - Nondestructive Testing and Health Monitoring of Ceramic Composites
Abstract no. 1915

# Combining in-situ synchrotron X-ray microtomography and acoustic emission to characterize damage evolution in ceramic matrix composites

Presenting author: Dr MAILLET Emmanuel
Affiliation: GE Research
1 Research Circle,12309 Niskayuna (NY, United States)
Co-authors: SINGHAL Anjali (GE Research), HILMAS Ashley (University of Michigan), GAO Yan (GE Research), ZHOU Ying (GE Research), HENSON Grant (GE Research)

In-situ synchrotron X-ray microtomography and acoustic emission (AE) were combined to study the behavior of ceramic matrix composite laminates during room-temperature mechanical testing. A detailed characterization of damage initiation and progression is obtained from microtomography, and the relationship between damage and AE is directly observed. A graphical representation of AE data, which has potential for real-time use, is employed to reveal differences in damage progression due to fiber architecture or loading mode. In addition, strong empirical relationships are observed between matrix crack area and AE energy, as well as between fiber breaks and number of AE events. This paper is based upon work supported by AFRL under Contract F865011C5227. Any opinions, findings and conclusions or recommendations expressed in this material are those of the author(s) and do not necessarily reflect the views of the USAF/AFMC Air Force Research Laboratory.

Topic: 12 - Nondestructive Testing and Health Monitoring of Ceramic Composites
Abstract no. 2199

# Imaging of Boron Nitride in Ceramic Matrix Composites

Presenting author: Dr TAYLOR Michael
Affiliation: Phoenix LLC
2555 Industrial Drive,53713 Monona (WI, United States)

Co-authors: OJARD Greg (United Technologies Research Center)

Ceramic matrix composites have three distinct phases: fiber, matrix, and an interface coating. For the current state of the art SiC/SiC composites, the means to differentiate the material from monolithic SiC is the presence of the weak interface which increases the toughness of the material. At present, the use of boron nitride to create this weak interface is the evolving standard for ceramic matrix composites. The means to deposit boron nitride onto the fibers is by chemical vapor infiltration. This distribution is assumed to be uniform within the material. Initial work performed has shown that 2D neutron radiography can detect the presence of boron nitride in ceramic matrix composites. In addition, the work has shown that there is a non-uniform distribution in some ceramic matrix composites based on the deposition approach. This fundamental initial work was expanded by utilizing neutron radiography inspection into additional directions with the intent of moving towards neutron computed tomography. The effort can be contrasted against x-ray radiography and x-ray computed tomography. These approaches and results may become useful for better modeling of ceramic matrix composite performance, both mechanical and thermal.

Topic: 12 - Nondestructive Testing and Health Monitoring of Ceramic Composites
Abstract no. 1909

# In situ X-ray microtomography characterisation of mechanical damage and failure in plain weave SiC/SiC composites


Presenting author: Prof ZHANG Daxu
Affiliation: Shanghai Jiao Tong University
A508 Mulan Building,800 Dongchuan Road,200240 Shanghai (China)

Co-authors: LIU Hailong (Shanghai Jiao Tong University), QI Heyin (Shanghai Jiao Tong University), WU Haihui (Aero Engine Corporation of China Commercial Aircraft Engine Co. Ltd), GUO Hongbao (Aero Engine Corporation of China Commercial Aircraft Engine Co. Ltd), HONG Zhiliang (Aero Engine Corporation of China Commercial Aircraft Engine Co. Ltd), CHEN Chao (Northwestern Polytechnical University), ZHANG Yi (Northwestern Polytechnical University)



The damage evolution and failure mechanism of plain weave SiC/SiC ceramic matrix composites were investigated by the in situ nano X-ray computed tomography (CT) experiment. The microstructure and initial defects of the material were obtained using a scanning electron microscope image. In order to acquire high resolution images, a mini-loading instrument was developed to perform in-situ X-ray CT tests within a laboratory nanofocus X-ray CT. Three dimensional image information of the materials under various tensile loading were reconstructed using the raw X-ray CT data. The damage modes and their developments were observed by examining and comparing the CT images of the in-situ tests under different tensile loading levels. The damage modes mainly include transverse matrix cracking, longitudinal matrix cracking, delaminations, fibre ruptures and fibre pullouts. A meso-scale progressive damage mechanism of plain weave SiC/SiC ceramic matrix composites was developed. It can be found from the in situ experiments that the uniaxial tensile stress-strain curves exhibited strong nonlinear characteristics. The initiation and development of damages occurred at the nonlinear phase. Transverse matrix cracks took place first, and then developed progressively with the increase of the tensile force. When the load reached a certain level, inter-tow cracks occurred and spread to large regions. Finally, fibre tows ruptured and catastrophic failures occurred. Close-ups of micro matrix cracks were observed after the material's failure. Many long fibre pullouts were found at the fracture surfaces of fibre tows.

Topic: 12 - Nondestructive Testing and Health Monitoring of Ceramic Composites
Abstract no. 2445

# Damage analysis of the elastic modulus of the composites by stitching diameter


Presenting author: Mme QIAO Zhiwei
Affiliation: SINOMA SCIENCE&TECHNOLOGY CO.,LTD
No. 198 Tongtian Road,Jiangning Science Park,211100 Nanjing (Jiangsu, China)
Co-authors: HU Fangtian (SINOMA SCIENCE&TECHNOLOGY CO. LTD), ZHU Jianxun (SINOMA SCIENCE&TECHNOLOGY CO. LTD)



A three-dimensional finite element model with different stitching diameter was established based on carbon fabric with 8/5 warp satin weave. Prediction of elastic modulus of composites by three-dimensional finite element model with total damage and local damage. The thicker the stitching line, the lower the tensile modulus of the composites, and the smaller the stitching line, the smaller the influence on the internal elastic modulus of the composites. The elastic modulus of composites with different diameters is less than 9.6% compared with that without stitching.Comparing with the actual elastic modulus of composites. The results show that the experimental results were larger than the finite element prediction, and the experimental result agrees with prediction.

Topic: 12 - Nondestructive Testing and Health Monitoring of Ceramic Composites
Abstract no. 1645

# Mechanical properties and failure mechanisms of 2D SiC/SiC composite with in-situ grown


Presenting author: M ZHAO DONGLIN
Affiliation: Northwestern Polytechnical University
127 West Youyi Road,710072 XI'AN (SHAANXI SHENG, China)

Co-authors:



Ceramic matrix composites with weak interphases show pseudo-plastic behaviors under tensile and shear loadings. In this work, an in-situ grown PyC interphase was prepared on the surface of amosicTM SiC fibers by chemical vapor reaction method. And then satin woven SiC fiber reinforced silicon carbide matrix composite (2D SiC/SiC) was prepared by chemical vapor infiltration (CVI) process. The mechanical properties of that composite were measured and the corresponding failure mechanisms were characterized. Results show that the tensile strength is about 666.52±30.90 MPa, the tensile modulus is 243±7.43 GPa, the compressive strength is about 807.31 MPa, the compressive modulus is 253.58 GPa, the flexural strength is 829.83±115.71 MPa, the flexural modulus is 155.55±2.99 GPa, and the interlaminar shear strength is 21.46±3.25 MPa. Compared with the plain woven SiC/SiC composite with BN interphase prepared by chemical vapor infiltration, the tensile, flexural, and compressive strength increases by 127%, 31%, and 51% respectively. Matrix cracking, crack deflection at the PyC interface, and fiber bridging are the critical failure mechanisms to control their pseudo-plastic behaviors. The great improvement of the above mechanical properties is ascribing to the increase of fiber volume fraction and the decrease of interface sliding stress.

Topic: 12 - Nondestructive Testing and Health Monitoring of Ceramic Composites
Abstract no. 2391

# Hole bearing properties and damage mechanisms of 2D C/SiC composites prepared by chemical vapor infiltration

Presenting author: Dr XUQIN Li
Affiliation: School of Materials Engineering, Chengdu Technological University, Chengdu, Sichuan 611730, P. R. China
No.1, Section 2,Zhongxin Avenue, Pidu,611730 Chengdu (Sichuan, China)

Co-authors:

As the development of large-scale complex components with all C/SiCs, mechanical joining and integration become an efficient and indispensable way for C/SiC applications, such as the body flaps of X38. Different from metallic mechanical joints, drilling a hole on the C/SiC plate not only cut the fibers off, but also introduce damages to the matrix and the interphase. Further, the hole surface is so rough that the corresponding stress distributions may result in several joint failure modes. In this work, plain woven C/SiC was prepared by chemical vapor infiltration (CVI) process. And a hole was drilled by a diamond boring bit. The effects of geometrical parameters on the hole bearing properties of the 2D C/SiC composite were then investigated. Results show that ductile crushed failure mode occurs when the ratio of width to hole diameter is larger than 3.47. Otherwise, net-tension failure mode happens. The porosity around the hole significantly influences the failure modes, that is, bearing failure mode occurs when the porosity is below a critical value, and shear-out failure mode may occur when the porosity increases to another critical value. The highest bearing strength is 477.0 MPa, which is ascribing to the coupled interaction between matrix cracking and fiber bridging mechanism. Crack deflection at the PyC interface and fiber bridging mechanism controls the ductile crushing process.

Topic: 12 - Nondestructive Testing and Health Monitoring of Ceramic Composites
Abstract no. 2093

# Determination of the stiffness matrix of ceramic matrix composites by ultrasonic time-of-flight measurements


Presenting author: Dr NEUBRAND Achim
Affiliation: Fraunhofer Intitute for the Mechanics of Materials
Woehlerstr 11,79108 Freiburg (Germany)

Co-authors: NEUBRAND Achim (Fraunhofer Intitute for the Mechanics of Materials Freiburg), HOHE Joerg (Fraunhofer Intitute for the Mechanics of Materials)



Knowledge of the complete stiffness tensor of a CMC material is a prerequisite for the simulation of mechanical behavior of CMC components. For this purpose, nine (for an orthotropic composite) respectively five (for a transverse isotropic composite) independent elastic constants need to be determined. Usually these constants are determined by determining stress-strain relationships in a set of mechanical tests followed by an inverse fitting procedure. These require the application of different loads (tensile, shear) in different directions – this is only possible using specimens with different geometry and orientation. This contribution describes a non-destructive ultrasonic time-of-flight measurement which facilitates the determination of the stiffness matrix of a composite using a single plate shaped specimen. Unlike in mechanical tests, there is no load applied to the specimens, i.e. a single specimen will yield consistent results in repeated measurements. This makes the presented method particularly suitable for monitoring property changes (e.g. due to ageing or fatigue). For the time-of-flight measurement, the CMC plate is immersed in a fluid and subjected to an ultrasonic pulse under variable angles of incidence and the pulse is detected by a receiver after passing through the CMC plate. The propagation velocity of the longitudinal and shear ultrasonic waves travelling through the specimen is then calculated for each angle of incidence from the time of flight of the acoustic pulses. The acquired propagation velocity data can be used to fit a complete set of elastic constants in an orthotropic material. In the present contribution, the stiffness tensors of ceramic matrix composites will be compared to experimental data obtained by conventional methods and the quality of the data obtained and possible reasons for differences will be critically discussed.

Topic: 12 - Nondestructive Testing and Health Monitoring of Ceramic Composites
Abstract no. 1751

# Load distribution of 2D C/SiC z-pinned joints prepared by chemical vapour infiltration


Presenting author: M LIU XIAOYING
Affiliation: Northwestern Polytechnical University
West Street,710072 XI'AN (SHAANXI SHENG, China)

Co-authors:



In this study, the 2D C/SiC z-pinned joints with a single pin or multiple pins were prepared by the online z-pinning method via chemical vapor infiltration. Their load distributions were studied via their load-displacement curves and corresponding strain-displacement curves. Results showed that: (a) The joints with multiple pins in longitudinal array and the joint with two pins in transverse array shows uniform load distribution ability due to the same shear strength as the joint with a single pin. (b) The joint with three pins in transverse array, the joint with three pins in triangle array and the joint with four pins in rectangle array show nonuniform load distribution among their z-pins due to their shear strengths are lower than the joint with a single pin. The nonuniform load distribution phenomenon may be attributable to the nonuniform microstructures such as the densities of z-pins at the joining region. (c) Oxidation damages aggravate the non-uniform load distribution among pins in transverse array, while does not change the uniform load distribution among pins in longitudinal array. It shows that the large-scale fiber bridging mechanism, which occurred after oxidation, benefits the uniform load distribution among pins in transverse array.

Topic: 12 - Nondestructive Testing and Health Monitoring of Ceramic Composites
Abstract no. 1947

# Tensile testing Ceramic Matrix Composites with in-situ Computed Tomography and Acoustic Emissions for real time crack detection and imaging

Presenting author: M BADRAN Aly
Affiliation: University of Colorado Boulder
UCB 426 Room 30,80309 Boulder (Colorado, United States)

Co-authors: MORSCHER Gregory (University of Akron), CREVELING Peter (University of Utah), BALE Hrishikesh (Carl Zeiss X-ray Microscopy), CZABAJ Michael (University of Utah), MARSHALL David (University of Colorado Boulder)

Two non-destructive experimental approaches to study damage and crack propagation in Ceramic Matrix Composites (CMCs) will be presented. Acoustic Emission (AE) and Computed Tomography (CT) were used simultaneously to examine a unidirectional CMC mini-composite while being loaded axially in tension. The AE transducers were placed on the grips at either end of the test section and the locations of cracks were determined by calculating the difference of travel times of acoustic waves. These locations were used to guide the locations of the CT scans to the regions of interest. After the experiment, the data were analyzed to assess the accuracy of matching the AE and CT locations. Image processing and segmentation were conducted to better identify and visualize cracks in 2D and 3D. We will report on the findings of this study including the methods of location matching, image processing and segmentation tools and future recommendations on how to setup AE and CT experiments.

Topic: 12 - Nondestructive Testing and Health Monitoring of Ceramic Composites
Abstract no. 2301

# Electrical resistance methodologies for damage identification in CMCs

Presenting author: Prof MORSCHER Gregory
Affiliation: University of Akron
Mechanical Engineering Department,44325 Akron (OH, United States)

Co-authors: SINGH Yogesh (University of Akron)

Electrical resistance has been used to effectively monitor damage accumulation whether distributed damage or crack growth in SiC, Si and C containing ceramic matrix composites. Since the fibers and matrix constituents for most non-oxide resistance has the potential to be an effective inspection technique for the purpose of quality control and/or structural health monitoring (SHM). Recent work using the relationship between in-plane and out-of-plane electrical resistivity and interaction with flaws has led to approaches useful for discerning interlaminar types of damage as well as other constituent/damage/structure non-uniformities. These findings will be discussed as well as the potential for implementing such approaches for quality control and SHM.

Topic: 12 - Nondestructive Testing and Health Monitoring of Ceramic Composites
Abstract no. 2390

# Modeling shear failure mechanisms of 2D C/SiC composite under off-axis loadin


Presenting author: Dr YI Zhang
Affiliation: Science and Technology on Thermostructural Composite Materials Laboratory, Northwestern Polytechnical University
West Youyi Rd.,No. 127,710072 Xi'an (Shaanxi, China)

Co-authors:



Crack deflection is one of the critical failure mechanisms of ceramic matrix composites. It is observed that the in-plane shear cracks always propagate perpendicularly to the fiber orientation no matter how the shear load direction changes, and fiber bridging mechanism controls the in-plane shear behaviour. In this study, plain woven carbon fiber reinforced silicon carbide matrix composite (2D C/SiC) was prepared by chemical vapor infiltration (CVI) process. The in-plane shear behaviours and failure mechanisms of 2D C/SiC composite were investigated under off-axis loading. A representative volume cell (RVC) is built to analyse the corresponding progressive damage behaviours. Results show that the in-plane shear modulus and strength increase with the increase of the angle between the fiber orientation and the loading direction, while the shear strain is in inverse proportion to the angle. The matrix crack involves via an inclined periodical mode under shear stress, just like the formation of "shear bands" in metals manufacturing processes. And the fibers are bended so as to bridge the matrix cracks, instead of breaking up. From the micromechanics view, the in-plane shear strength of 2D C/SiC is characterized in terms of periodical matrix cracking stress and fiber bridging stress. The progressive damage model can reproduce the nonlinear shear behaviours and shear damage mechanisms.

Topic: 12 - Nondestructive Testing and Health Monitoring of Ceramic Composites
Abstract no. 1715

# Modeling nonlinear shear behaviors of 2D C/SiC z-pinned joint


Presenting author: M CHEN Chao
Affiliation: Northwestern Polytechnical University
West Youyi Rd., No. 127,710072 Xi'an (Shaanxi, China)
Co-authors: ZHANG Yi (Northwestern Polytechnical University), ZHANG Litong (Northwestern Polytechnical University), CHENG Laifei (Northwestern Polytechnical University), LIU Xiaoying (Northwestern Polytechnical University), ZHAO Donglin (Northwestern Polytechnical University)



The effects of total porosity of 2D C/SiC z-pin were studied on the tensile behaviors and the failure mechanisms of 2D C/SiC z-pinned joint with a single pin. Results showed that: (a) When the joint is under tension, the 2D C/SiC z-pin is sheared off under the coupled shear and bending stress. And the interface sliding and fiber bridging mechanisms control the fracture process of z-pin. (b) As the total porosity of 2D C/SiC z-pin increases, the joint shear strength decreases according to a power law. A modified rigid body sliding model is proposed to quantitatively characterize the relationship between the total porosity of 2D C/SiC z-pin and the shear strength of 2D C/SiC z-pinned joint. It shows that the shear strength of 2D C/SiC z-pinned joint equals the in-plane shear strength of 2D C/SiC composite plus the bending stress component of the fiber bridging stress. (c) A nonlinear finite element model for 2D C/SiC z-pinned joint is developed to accurately predict its nonlinear tensile behaviors and the shear rupture process of 2D C/SiC z-pin. The prediction shows that the joint nonlinear tensile behaviors are controlled by the coupled shear and bending failure process of 2D C/SiC z-pin. Hence, the calculated results verify the modified rigid body sliding model.

# Topic: 13

# Joining & integration

Topic: 13 - Joining & integration
Abstract no. 2257

# Active Brazing of Diboride and Carbide-based Ceramics and Ceramic-Matrix Composites


Presenting author: Dr ASTHANA Rajiv
Affiliation: University of Wisconsin-Stout
School of Engineering,54751 Menomonie (Wisconsin, United States)
Co-authors: SINGH Mrityunjay (Ohio Aerospace Institute Cleveland Ohio USA)



Silicon carbide and zirconium diboride-based ceramics were used to create self-similar joints and dissimilar joints with metallic substrates such as copper-clad-molybdenum, titanium, and nickel-based alloys using Ag, Ti, Ni, and Pd-base metallic brazes with liquidus temperatures in the range 815 to 1240?C. The joints were characterized using field emission scanning electron microscopy (FESEM), energy dispersive spectroscopy (EDS), and Knoop microhardness test. Joint integrity was influenced by chemical interactions with the molten braze, surface preparation, and braze composition and properties. Both low- and high-temperature active brazes formed metallurgically sound joints. The high-temperature Pd-base brazes formed the most complex multilayer interfaces due to substrate and fiber dissolution, chemical reactions, and melt infiltration and led to some tendency for fiber ply delamination at the bonded interface due to large CTE mismatch and low inter-laminar shear strength. Knoop hardness scans across joints were reproducible and consistent with the nature of the substrates and reaction zones. The brazes considered may be suitable to join components outside of the hottest zones especially in physical locations that are amenable to external cooling. Potential thermal benefits of these brazed joints for enhanced heat transfer are highlighted and recommendations to mitigate the residual stresses are proposed.

Topic: 13 - Joining & integration
Abstract no. 2259

# Study on shear property of C/SiC rivets made of different preforms


Presenting author: M LIU Xiaochong
Affiliation: Northwestern polytechnical university
PO BOX 547#,West Youyi Rd. NO.127,710072 Xi'an (shaanxi, China)

Co-authors: LIU Chidong CHENG Laifei (Northwestern polytechnical university)



Rivets play a key role in structural reliability and functional achievement for the whole component. This paper systematically investigates the shear properties of the ceramic matrix composite rivets made of 2D and the ones made of a quasi-1D preform.It is found that the shear property of 2D rivets strongly depend on the angle between the laminated direction of preform and the shear load direction, quasi-1D rivets have a higher shear performance and narrow dispersion than the 2D ones. Furthermore, this paper revealed the mechanism to understand the excellent performance of quasi-1D rivets.

Topic: 13 - Joining & integration
Abstract no. 2157

# Fabrication and joining technology development of SiCf/SiC composites cladding


Presenting author: Dr LEE Hyeon-Geun
Affiliation: Korea Atomic Energy Research Institute
989-111 Daedeok-daero,Yuseong-gu,34057 Daejeon (South Korea)

Co-authors: KIM Daejong (Korea Atomic Energy Research Institute), PARK Ji Yeon (Korea Atomic Energy Research Institute), KIM Weon-Ju (Korea Atomic Energy Research Institute)



SiC ceramics and composites are considered as structural materials for fusion and fission system due to its excellent mechanical properties at high temperature and good irradiation tolerance. SiC fiber reinforced composites tube is one of the candidate fuel cladding for Light Water Reactor (LWR) within the Accident Tolerant Fuel (ATF) design concept. We have developed duplex and triplex structured SiC composites tube that can be applied with ATF cladding for LWR by chemical vapor infiltration method. 30 cm long SiC composites tubes were uniformly manufactured using large size CVI reactor and high density SiC composites tubes were fabricated by applying densification technology using SiC nanowire. Hoop strength, thermal shock resistance, gas tightness, hydrothermal corrosion resistance and high temperature vapor corrosion resistance of the SiC composites tubes were characterized by each suitable equipment. In order to use SiC composites cladding as ATF cladding, it is very important to develop joining technology between SiC composites cladding and SiC end cap. The brazing materials containing heterogeneous element like as Ti, Al usually have been used as an interlayer for the joining of SiC ceramics. In this study, we used the interlayer, which is SiC amorphous layer and Si layer deposited by PVD method, composed of the same elements as the SiC cladding and the end cap. The joining between CVD SiC tubes and CVD SiC, between SiC composites tubes and CVD SiC were successfully performed through the heat treatment with a high joining strength between the tube and the end cap. The microstructure of the joint were characterized by SEM and TEM. The corrosion resistance and the gas tightness of the joint were also analyzed.

Topic: 13 - Joining & integration
Abstract no. 1985

# Functionalizing ceramic matrix composites via an integrated metallic sub-structure with comparable feature size.


Presenting author: Dr APOSTOLOV Zlatomir
Affiliation: US Air Force Research Laboratory
2807 Liga Ct,45430 Beavercreek (Ohio, United States)

Co-authors: HECKMAN Elizabeth (US Air Force Research Laboratory), CINIBULK Michael (US Air Force Research Laboratory)



Due to the extreme nature of both their processing and applications, imparting functional properties to high-temperature ceramic matrix composites (CMCs) is by default a challenging prospect. With this class of materials finding increasingly wider applications, however, the need for corresponding structural state awareness is growing accordingly, and as such is probably the single most important example of added functionality requirements. There are two general approaches to address this challenge – use the features of the native constituents of the CMC as they evolve during degradation, or try to integrate additional phases with characteristics more convenient or fitting to the specific phenomenon of interest. This work focuses on the latter of the two approaches, by illustrating some of the processing considerations which are encountered upon integrating a refractory metal-based sub-structure within a polymer infiltration and pyrolysis-derived CMC with comparable feature size. The focus in particular, is on ensuring the retention of the metallic character of the sub-structure by investigating the reactivity between the metal and the CMC constituents, while being able to complete the full process of matrix densification. The roles of composition, time, temperature, and the morphology of the interaction volume during processing are evaluated, and preliminary treatment profiles for several different metal-CMC combinations are proposed.

Topic: 13 - Joining & integration
Abstract no. 2306

# Joining and mechanical testing of oxide/oxide ceramic composites


Presenting author: Prof FERRARIS MONICA
Affiliation: POLITECNICO di Torino
DISAT, corso Duca degli Abruzzi 24,corso Duca degli Abruzzi 24,10129 TORINO (TO, Italy)
Co-authors: "AKRAM Muhammad Yasir ( POLITECNICO di Torino), CASALEGNO Valentina ( POLITECNICO di Torino), PUCHAS Georg ( 2. Department of Ceramic Materials Engineering, University of Bayreuth, Bayreuth, Germany), KRENKEL Walter ( 2. Department of Ceramic Materials Engineering, University of Bayreuth, Bayreuth, Germany), ROSZEITIS Sven ( Fraunhofer Institute for Ceramic Technologies and Systems IKTS, Germany), MARTIN Hans-Peter ( Fraunhofer Institute for Ceramic Technologies and Systems IKTS, Germany)



Efficient joining materials and techniques are of critical importance for the integration of CMCs in high performance structures. Continuous NextelTM 610/alumina-zirconia composites were successfully joined to themselves and to metal alloys by using several glass-ceramics and brazes. Single lap off-set shear tests and four-point bending tests were performed at room temperature and at 850 °C to investigate the mechanical strength of the most promising joints. Thermal ageing was performed at 850 °C for 100 h in air to evaluate the thermal stability of the joined components. The results showed that the glass-ceramic joints were oxidation resistant and the joined interfaces remained unchanged after these oxidation tests. Single lap off-set shear tests on joined samples resulted in a delamination of the composites. The average flexural strengths of the glass-ceramic joined samples were 71 MPa and 81 MPa, at room temperature and at 850 °C, respectively.

Topic: 13 - Joining & integration
Abstract no. 2171

# Ytterbium disilicate-based glass ceramic as joining material for ceramics and CMCs


Presenting author: Prof SALVO Milena
Affiliation: Politecnico di Torino
Corso Duca degli Abruzzi 24,10129 Torino (Italy)

Co-authors: D'ISANTO Fabiana (Department of Applied Science and Technology (DISAT), Politecnico di Torino Turin Italy), SMEACETTO Federico (Department of Applied Science and Technology (DISAT), Politecnico di Torino Turin Italy)



Since the production of complex and large structures is difficult to be performed, a critical issue for a wider use of ceramics and ceramic matrix composites (CMCs) is the development of inexpensive and reliable joining methodologies to combine simple components into more complex ones.

Among the proposed joining materials for ceramics and CMCs, glass-ceramics have challenging perspectives, thanks to their versatility in compositions and tailorable properties (i. e. thermal expansion coefficient, softening and glass transition temperatures).

This study describes the development of a glass-ceramic joining and coating material for SiC and Si3N4 and SiC based composites. The glass-ceramic contains an ytterbium disilicate phase embedded in a barium aluminium boron silicate glass. The glass-ceramic was obtained by in situ reaction of Yb2O3 particles with the silica of the glass matrix. The reaction between the ytterbium oxide and the barium aluminium boron silicate glass matrix by viscous phase is reviewed and discussed. Coated samples and sandwich-like joined specimens were prepared by means of the slurry technique and a subsequent heat treatment with no external pressure being applied. Morphological and compositional analyses of the joints were carried out by scanning electron microscopy (SEM) and energy dispersive X-ray spectrometry (EDS) before and after mechanical tests.

Topic: 13 - Joining & integration
Abstract no. 2149

# Electron current assisted direct joining of low resistivity grade silicon carbide


Presenting author: Dr KOYANAGI Takaaki
Affiliation: Oak Ridge National Laboratory
1 Bethel Valley Road,37831 Oak Ridge (TN, United States)

Co-authors: LI Huaxin (1Hefei University of Technology), ANG Caen (University of Tennessee Knoxville), KATOH Yutai (Oak Ridge National Laboratory)



Conventional direct joining of SiC fiber reinforced SiC matrix composites typically requires high temperature (>1900°C), high pressure (>10 MPa), and a holding time of ~1 hour, which makes this method practically inapplicable because of SiC fiber degradation under such an environment. The overarching objective of this study is advancement of direct joining technology for SiC materials by electron current assisted (ECA) method. Previous studies on ECA sintering and joining of SiC with various forms suggested the possibility of reducing the temperature and time of the direct joining. To demonstrate ECA direct joining, low resistivity grade, high purity SiC made by chemical vapor deposition was joined without an interlayer. Two types of experimental setting were used in this study: ECA joining with and without using a graphite die. The setting with the graphite die results in the electron current primarily passing through the graphite die because of the relative conductivity. In contrast, the setting without the graphite die allows effective Joule's heating of the SiC specimens. Typical temperature ramp rate and holding time were 100°C/min and 10 min, respectively. Successful joining was demonstrated at 1700°C without the graphite die. In contrast, the joining was incomplete with the graphite die. The results suggest that joule heating of the specimen itself was effective for successful joining. Microstructures of the specimens joined under different experimental conditions were analyzed by electron backscatter diffraction. The underlying joining mechanism will be discussed based on the microstructures.

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Topic: 13 - Joining & integration
Abstract no. 2159

# Wetting and phase interaction between Cf/SiC and transition metal disilicides-based alloys


Presenting author: Dr TATARKO Peter
Affiliation: Institute of Inorganic Chemistry, Slovak Academy of Sciences
Dubravska cesta 9,84536 Bratislava (Slovak Republic)

Co-authors: VALENZA Fabrizio (Institute of Condensed Matter Chemistry and Energy Technologies - ICMATE National Research Council –CNR Via De Marini 6 161-49 Genoa Italy), UNSAL Hakan (Institute of Inorganic Chemistry Slovak Academy of Sciences Dubravska cesta 9 845 36 Bratislava Slovakia), KOVALCIKOVA Alexandra (Institute of Materials Research Slovak Academy of Sciences Watsonova 47 40 1 Kosice Slovakia), BYSTRICKY Roman (Institute of Inorganic Chemistry Slovak Academy of Sciences Dubravska cesta 9 845 36 Bratislava Slovakia)



The aim of the work was to investigate the wetting behaviour and infiltration phenomena, which occur during reactive melt infiltration (RMI) or coating of different types of Cf/SiC composites. The wetting and phase interaction of molten transition metal disilicides (ZrSi2 and HfSi2) was first investigated in different atmospheres (vacuum and Argon) at 1670°C. It was found out that a significant loss of Si from the CMC substrates caused an increase of the melting temperature and suppressed the spreading of the alloy over the surface of CMCs when tested in vacuum. On the other hand, wetting behaviour and spreading of the alloy was improved in argon atmosphere. The formation of ZrC phase with a high melting point partially hindered the spreading of the melt over the surface of CMCs. Interestingly, the wetting and infiltration of the alloy was improved for the Cf/SiC composites manufactured using a higher number of PIP cycles (Polymer Infiltration and Pyrolysis) when compared to the composite with less PIP cycles. The wetting and material interaction between Cf/SiC substrates and various different transition metal disilicides-based alloys (containing B4C, and/or RE-additives) was then investigated. A thorough phase analysis, such as micro-XRD and SEM, and theoretical predictions helped to understand the interactions between the surface and the alloy when preparing a ZrB2(HfB2)-SiC-RE coating layer on the surface of Cf/SiC.

Topic: 13 - Joining & integration
Abstract no. 2075

# Mechanical characterization of surface engineered CMCs joints


Presenting author: Prof CASALEGNO Valentina
Affiliation: POLITECNICO DI TORINO-DISAT
Corso Duca degli Abruzzi,24,10129 Torino (Torino, Italy)

Co-authors: VALENZA Fabrizio (National Research Council – Institute of Condensed Matter Chemistry and Technologies for Energy (CNR-ICMATE), BALAGNA Cristina (Politecnico di Torino-DISAT), SALVO Milena (Politecnico di Torino-DISAT), FERRARIS Monica (Politecnico di Torino-DISAT)



This work presents the surface engineering with brazing to join SiC/SiC composites and to improve the joints strength. Surface engineering was carried out through the Selective Thermal Removal (STR) of SiC fibres from the SiC/SiC composite to obtain "brush-like" surfaces; the modified composites were then joined by means of a AgCuTi braze alloy. In order to to investigate if a "brush-like" interface could enhance the mechanical strength of the joint by increasing the mechanical interlocking with the brazing alloy at the micron scale, the joints with and without surface engineering were tested by single lap offset in compression.

The brush-like SiC/SiC interface were well infiltrated by the Ag-based brazing alloy, able to give a composite-like fracture surface and a "plastic-like" load/displacement curve after single lap offset test in compression of the joints.

At this stage of the research, the STR process seems to be effective in increasing the joint toughness, but the treatment must be modified in order to avoid degradation of the whole SiC/SiC. Further experimental activity will focus on localised and less harsh thermal process, in order to find a good compromise between surface engineering and composite integrity.

Topic: 13 - Joining & integration
Abstract no. 2277

# Gas-Tightness of Joint Oxide Ceramic Matrix Composite Tubes at High Temperatures


Presenting author: M SCHMIDT Jens
Affiliation: Fraunhofer ISC
Gottlieb-Keim-Straße 62,95448 Bayreuth (Germany)

Co-authors: ECKARDT Christian (Fraunhofer ISC), SCHULTE Rüdiger (Fraunhofer ISC)



Oxide ceramic matrix composites (OCMC) show high mechanical strength > 300 MPa and deformation up to 0.5 %. Due to their high porosity of about 30-40 vol.-% OCMC are not gas tight. However, the material can only be utilized for liquid or gas transportation if the required tightness is achieved. Additionally, for industrial pipeworks suitable joinings are needed since the manufacturing through winding or lay-up of fabrics is limited to tubes with open ends. Both, simple tubes as well as joint tubes must be highly dense and have to withstand thermal shock during heating up and cooling down cycles. In order to improve the gas tightness the open porosity of the matrix must be partly or fully closed without losing its ductile performance. In this research work the impregnation of the matrix was accomplished by using high viscosity glass solders with the addition of ceramic particles as filler. Two different joining designs, the tube-to-tube joining as well as the tube-to-flange joining were investigated. In order to reach high strength in the joints the form closure was designed. By increasing the surface of face-to-face joined specimens the strength could be increased considerably. Mechanical characterization of the flat joints was conducted by shear strength measurements acc. to DIN EN 1894. The strength of joints was determined by using two different self-designed test benches. The helium leakage rate was determined according to standard DIN EN 1779. A steel tube with a dummy flange and full gas-tightness was used as a reference part. The gas-tightness of the joint OCMC tubes was evaluated at room temperature and temperatures up to 1300 °C in a tube furnace. The homogeneity of the tubes and potential artefacts like pores or microcracks in the joint could be detected by microstructural investigations using SEM and non-destructive computer tomography.

Topic: 13 - Joining & integration
Abstract no. 1944

# High Temperature-Reactivity evaluation of Al-Ti alloys in contact with SiC

Presenting author: Dr DEZELLUS Olivier
Affiliation: Université Lyon 1
LMI - Batiment Chevreul,6 rue Victor Grignard,69100 Villeurbanne (France)

Co-authors: CHIRIAC Rodica (Université Lyon 1), TOCHE François (Université Lyon 1), PASSERONNE Alberto (CNR), MUOLO Maria-Luigia (CNR), CACCIAMANI Gabriele (University of Genoa), VALENZA Fabrizio (CNR), GAMBARO Sofia (CNR)

Several industrial processes interested in coupling SiC to Al-Ti alloys (e.g. metallization of SiC components, brazing of SiC parts or synthesis of SiC based composite materials) require a deep knowledge about Al-Ti/SiC interactions occurring at high temperatures. To this end, the surface reactivity between SiC and Al-Ti alloys (Al3Ti and (Al+Al3Ti) systems) has been analyzed by specific experiments (wetting, DSC, microstructural examinations) as well as by a thermodynamic approach (CALPHAD method).
An Al-C-Si-Ti thermodynamic database has been successfully set up to calculate several sections and projections on which a comparison was made possible of the computed, expected solid phases formed at interface with the ones characterized in wetting experiments. By this means, the evolution of liquid and solid phases has been interpreted and discussed, defining the Ti3(Al,Si)C2 mixed MAX-phase as the main interfacial product coming from the chemical reaction, as a function of temperature and alloy composition.
This work provides a guide for the choice of the operating parameters in processes such as brazing or SiC metallization in microelectronic applications, in which the control of the interfacial products is one of the most delicate production step. The proposed approach to follow the pathway of liquid composition with time and temperature during liquid/solid interaction, successfully applied to interpret the microstructure obtained in wetting experiments, is a promising way to interpret more complex cases such as homogeneous or even heterogeneous brazing processes.

Topic: 13 - Joining & integration
Abstract no. 2773

# Experimental study on the feasibility of using liquid Si-Ti alloys for joining CMC via reactive infiltration process

Presenting author: Dr GIURANNO Donatella
Affiliation: Institute of Condensed Matter Chemistry and Technologies for Energy, National Research Council, CNR-ICMATE, Genoa, Italy
Via De Marini 6,16149 Genova (Italy)

Co-authors: BRUZDA Grzegorz (Foundry Research Institute Krakow Poland), NOWAK Rafal (Foundry Research Institute Krakow Poland), POLKOWSKI Wojciech (Foundry Research Institute Krakow Poland), SOBCZAK Natalia (Foundry Research Institute Krakow Poland), NOVAKOVIC Rada (CNR-ICMATE Genoa Italy)

SiC-based composites are ideal candidates for highly demanding applications because of their outstanding thermo-mechanical response and corrosion resistance.
Reactive infiltration of Si-based alloys into C and SiC-C preforms might be a costless alternative to fabricate highly performant MMCs and CMCs composites and even to obtain reliable joints for assembling in largescale and complex-shaped components or for integration of CMC into metallic structures.
In order to optimize reactive infitration and to tailor the joint microstructures, the knowledge of intefactial phenomena in terms of wettability, reactivity, surface properties and thermodynamics concerning reacting phases, is a key contribution [1, 2].
Systematic investigation of wetting, reactivity, surface properties and thermodynamics of Si-Ti melts in contact with Glassy-C and HIP-SiC substrates has been previously carried out. Examples of CMC joined by Si-Ti alloys via reactive infiltration process will be reported and supported by a critical examination of developed microstructures and interfacial phenomena observed mainly related to the Ti-content and operating parameters. In particular, the Si-Ti/C and Si-Ti/SiC resulting interfaces will be carefully analyzed [3] and the overall phenomena occurred within the process will be deeply discussed.
Acknowledgements
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Abstract no. 2858

# Wetting behavior and reactivity of liquid Si-10Zr alloy in contact with glassy carbon and SiC


Presenting author: Dr GIURANNO Donatella

Affiliation: Institute of Condensed Matter Chemistry and Technologies for Energy, National Research Council, CNR-ICMATE, Genoa, Italy
Via De Marini 6,16149 Genova (Italy)

Co-authors: POLKOWSKI Adelajda (Foundry Research Institute Zakopia?ska 73 Str. 30-418 Krakow Poland.), POLKOWSKI Wojciech (Foundry Research Institute Zakopia?ska 73 Str. 30-418 Krakow Poland.), BRUZDA Grzegorz (Foundry Research Institute Zakopia?ska 73 Str. 30-418 Krakow Poland.), RICCI Enrica (National Research Council of Italy-Institute of Condensed Matter Chemistry and Technologies for Energy Via De Marini 6 16149 Genova Italy.), NOVAKOVIC Rada (National Research Council of Italy-Institute of Condensed Matter Chemistry and Technologies for Energy Via De Marini 6 16149 Genova Italy.)



Currently, the main challenges facing the development of advanced MMCs and CMCs are related to the costly fabrication of tailored and performant interfaces microstructures for highly demanding applications, to optimize the fabrication process and to identify ad-hoc the metal matrix for each application.

In designing advanced refractory composites, reactive infiltration of liquid Si-enriched Si-Zr alloys into C- or SiC-based preforms might be a cost-less alternative.

Fundamental investigations on interfacial phenomena between liquid Si-Zr alloys in contact with C and SiC substrates, are key steps for optimizing liquid assisted processes (reactive infiltration). Targeted wettability and reactivity studies can easily provide useful indications for solving many technological problems affecting the reactive infiltration mechanisms, such as pore closure/narrowing phenomena.

Aiming to ‘‘mimic’’ the conventional operating conditions imposed, the contact heating sessile drop method was applied to better understand of the interaction phenomena occurring between the liquid Si-10%atZr alloy in contact with glassy carbon and SiC substrates. Specifically, the contact angle values as a function of time were measured over the temperature range of T = 1354-1500°C under an Ar atmosphere.

The interaction phenomena observed in terms of wettability and spreading kinetics are temperature and time-dependent.

The kinetics of SiC crystal growth at the interface was analyzed and related to the processing parameters (i.e. T and t). In both cases, the SiC crystals growth and their packaging phenomenon, is favored at higher temperatures and longer time of contact with the substrate.

At the Si-10%atZr alloy/GC and Si-10%atZr alloy/SiC intefaces, reactive and no-reactive wetting mechanisms, respectively, were observed.

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Topic: 13 - Joining & integration
Abstract no. 2308

# Joining of SiC/SiC for nuclear energy production


Presenting author: Prof FERRARIS MONICA
Affiliation: POLITECNICO di Torino
POLITECNICO TORINO - DISAT,corso Duca degli Abruzzi 24,10129 TORINO (TO, Italy)
Co-authors: CASALEGNO Valentina (POLITECNICO di Torino #NOM?), HERRMANN Marion (Technische Universität Dresden Dresden Germany), HINOKI Tatsuya (Kyoto University Japan), KOYANAGI Takaaki (Oak Ridge National Laboratory Oak Ridge USA), KATOH Yutai (Oak Ridge National Laboratory Oak Ridge USA)



Several materials have been proposed for joining of SiC/SiC components for nuclear fusion and fission reactors.
Among them, some glass-ceramics gave promising results as pressure-less joining materials for slurry- and laser-based joining technologies suitable for a nuclear environment.
For some of them, microstructure and interfaces with SiC have been investigated before and after neutron, and ion irradiation.
However, an additional key issue for joined and coated SiC-based materials in LWRs coolant environments is their hydrothermal corrosion.
The present work reports on several pressure-less joining options for SiC-based components: some refractory metals and glass-ceramics have been used to join SiC/SiC composites, then tested in autoclave at 320 °C, in high purity water, under 15 MPa pressure for 8 hours, as a pre-screening.
Some mechanical tests on the joined composites will be also discussed.


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Part of the research leading to these results has received funding from the European Union’s Horizon 2020 research and innovation programme under grant agreement No 740415 Project “IL TROVATORE - Innovative Cladding Materials for Advanced Accident-Tolerant Energy Systems”.

Topic: 13 - Joining & integration
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# INTERFACIAL REACTION KINETICS AND MICROSTRUCTURAL EVOLUTION OF C/SiC COMPOSITES TO TITANIUM ALLOY JOINTS


Presenting author: Mme TüLBEZ Simge
Affiliation: Phd. Student
Roketsan Industries Inc.,Ankara-Samsun Karayolu 40.Km,06780 Ankara (Turkey)
Co-authors: DERICIO?LU Arcan (1Department of Metallurgical and Materials Engineering Middle East Technical University)



Brazing of ceramic matrix composites and metal components is an emerging manufacturing technology in which a furnace or a heat source is used to join the materials using a brazing filler alloy. Brazing of ceramic and metal has received considerable attention in the fields of nuclear reactor, aerospace, automobile and electrical engineering.
In the present study, C/SiC composite and Ti6Al4V alloy are joined using Cusil (72 wt% Ag + 28 wt% Cu) and Cusil ABA (63 wt% Ag + 33.25 wt% Cu + 1. 25 wt% Ti) filler alloys by vacuum brazing method. In the scope of the study, the effect of active element on the performance of brazing was examined. Ceramic matrix composites are expected to reveal direct wetting by active metal containing filler materials promoting chemical reactions, which results in improved bonding characteristics. For this purpose, interfacial microstructure and formation mechanism of the brazed joints were studied. Moreover, the effect of brazing temperature on the microstructural evolution and mechanical properties of joints was investigated.
Microstructural characterization of the brazed joints was performed using optical microscope as well as Scanning Electron Microscope (SEM). XRD analyses were conducted to identify phases present in the C/SiC composite – Ti6Al4V alloy joints. Hardness and shear tests were conducted for the mechanical characterization of the brazed joints.

Topic: 13 - Joining & integration
Abstract no. 2307

# "RM-Wrap" joining technology for CMC

Presenting author: Prof FERRARIS MONICA
Affiliation: POLITECNICO di Torino -Italy
corso Duca Abruzzi 24,10129 Torino (ITALIA, Italy)

Co-authors: GIANCHANDANI Pardeep Kumar (1Mehran University of Engineering and Technology - Jamshoro Sindh Pakistan), DE LA PIERRE Stefano (POLITECNICO di Torino -Italy), CASALEGNO Valentina (POLITECNICO di Torino -Italy), SALVO Milena (POLITECNICO di Torino -Italy), DLOUH_ Ivo (Institute of Physics of Materials – Brno Czech Republic)

A novel joining technique "RM-wrap" (RM = Mo, Nb, Ta, W, Zr) has been successfully applied to join C/SiC and SiC/SiC to themselves and to ceramics. Optimized joining treatment consisted of heating to 1450 °C with heating rate of 1000 °C / hour followed by a dwell time of 5 minutes in Argon flow. The joints have been characterized by morphological analysis, microhardness and lap shear tests at room temperature and 1000 oC. The joining material is an in situ composite made of a Si matrix reinforced by metal disilicides (MoSi2, NbSi2, TaSi2, ZrSi2 …etc..). The joint morphology and elemental composition of the joining material have been investigated in detail using XRD, FESEM and EDS. A test system with vacuum and/or inert gas atmosphere chamber (Zwick/Roell-Messphysik-Maytec) has been used to measure the lap shear tests of the joints up to 1200 °C .

Topic: 13 - Joining & integration
Abstract no. 2517

# Functionally Graded energy recyclable ceramic-metal composites and its Applications


Presenting author: Prof KWON Hansang
Affiliation: Pukyong National University
45 Yongso-ro, Namgu,Eng.Bldg.1,48513 Busan (South Korea)
Co-authors: KIM Kyungju (Next Generation Materials Co. Ltd.)



A state-of-the-art solid-state lighting (SSL) device comprises a blue lightemitting device covered with a yellow-emitting phosphor. In a high-power SSL device, thermal stability is an essential requirement for the phosphor because the phosphor is constantly irradiated with high-power blue light, which causes thermal degradation. In this work, a thermally robust functionally graded materials (FGM) of ceramic-metal composite fused with low-melting glass is introduced.

The ceramic-metal FGM was successfully  well bonded without any generation of micro pore and crack during spark plasma sintering process.The FGM exhibits a high thermal conductivity of 31.6 W m?1 K?1 and the composite combined with a 4 W blue laser diode (LD) exhibits excellent thermal stability in luminous flux and chromaticity. Furthermore, energy is recycled in the FGM during lighting by coupling to a thermoelectric (TE) module. In the FGM-TE system, the output voltage and current reveal 289 mV and 77 mA, respectively, at an output of 430 lumens under a 4 W blue LD. The results provide a starting point for further research in the FGM design for energy recycling with high-power white lighting system.

# Topic: 14

# CMC Applications in Space Transportation

Topic: 14 - CMC Applications in Space Transportation
Abstract no. 2231

# CMC based Structures for Hypersonic Flight

Presenting author: M WEIHS Hendrik
Affiliation: DLR
Pfaffenwaldring 38-40,70569 Stuttgart (Germany)

Co-authors:

Future space transportation systems, especially with returnable elements for re-use, need different structural concepts compared to expendable concepts. Fiber reinforced ceramic materials were developed and improved to fit with future requirements for such reusable space vehicles. This includes besides the classical C/C-SiC and SiC/SiC CMC materials also ultra high temperature resistant ceramics, oxidation resistant oxide based ceramics (WHIPOX) and structural ceramic with adjustable porosity for acoustic damping and active effusion cooling (OCTRA). CMC structures are promising materials for Re-entry vehicles and exposed areas for hypersonic flight systems (e.g. Hexafly, Hifire 5B, SCRAMSPACE), Beside the classical thermal protection application, moveable and functional elements like flaps or deployable heat shield elements are key elements
A full scale re-entry test flight is expensive and needs in general a launch system with orbital capacity. To simplify and to reduce costs a step by step approach seems to be practicable and less risky. Thus, sounding rockets are a very attractive vehicle to perform related research. Thus, design concepts and structural elements for new space transportation systems were developed using sounding rocket structural subsystems as a reference to get the opportunity to test and demonstrate such structures in flight.
This paper gives an overview of related structural developments within DLR.

Topic: 14 - CMC Applications in Space Transportation
Abstract no. 2459

# MTG Star Tracker Assembly Bracket – A Successful Story of Fabrication light-weighted Structures


Presenting author: M KRÖDEL Matthias
Affiliation: Engineered Ceramic Materials GmbH
Am Bleichbach 12,85452 MOOSINNING (Germany)
Co-authors: KOMADINA Jonathon (OHB System AG), PERRIN Laurent (Thales Alenia Space France), BERILL Marc (European Space Agency)



2014 ECM received from OHB, Germany the contract for the design, fabrication, test and qualification of the Star Tracker Assembly Bracket for the next generation weather satellite (MTG - Meteosat Third Generation), developed by ESA and operated finally by EUMETSAT. In December 2018 ECM delivered the last two Flight models fully integrated and tested to OHB, Bremen. This project was for ECM a big challenge being for the first-time prime contractor of a small subsystem consisting of an assembly of metal components associated to our HB-Cesic® bracket structure.
In this paper we will report about the successful manufacturing process of seven flight models including STM Model for qualification in a rather short time frame of less than 2 years after CDR. This paper is focused on the successful manufacturing and precision machining of these complex light weighted structures with low tolerance requirements to demonstrate ECMs capability of fabricating such large ceramic components.

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Abstract no. 2078

# VINCI Engine Composite Nozzle Extension For Ariane 6

Presenting author: M COPERET Herve
Affiliation: ArianeGroup
Les Cinq Chemins,33185 LE HAILLAN (France)

Co-authors:

The Ariane 6 VINCI engine thermo-structural composite Nozzle Extension is designed and manufactured by ArianeGroup on its Le Haillan site in France. The Nozzle Extension channels the combustion chamber gas flow, sustains the thrust and provides the highest possible Isp with the minimum mass. The Nozzle Extension is roughly 2 meters high with an exit diameter of about 1.8 meters. It is composed of two radiative thermo-structural cones, the aft cone in Carbon/Carbon-Silicon Carbide and the forward cone in Carbon/Carbon with an oxidation protection. The A6 design has been simplified wrt to the A5ME design: it has only 2 cones (instead of 3) and is no longer deployable in flight. This allowed simplifying the junction between the 2 cones and reducing the mass and the recurring cost. As of today, five development hardware pieces have been manufactured within the programme ; three under the A5ME frame (S1 a fixed forward cone and two complete Nozzle Extensions NE1 and NE2) and two under the A6 frame (NE3 and NEQ). The first A6 flight Nozzle extension is available (FM1). Since 2006, S1, NE1 and NE2 have successfully participated in a total of seven full-scale altitude simulation test campaigns at the DLR test bench P4.1 and in three Engine Dynamic test campaigns, one in ArianeGroup Vernon site and two in IABG, Munich. The first full-scale altitude simulation test qualification campaign M6 at P4.1 with the first A6 Qualification Nozzle Extension NE3 was held successfully from March to July 2017. NE3 has seen more than four life durations in 7 tests. The dynamic qualification campaign EDQ at IABG with the second A6 Qualification Nozzle Extension NEQ was held successfully in June and July 2017. The second full-scale altitude simulation test qualification campaign Q1 with the second Qualification Nozzle Extension NEQ was held successfully from January to June 2018. NEQ has seen more than four life durations in 6 tests. The Qualification Review was held in February 2019.The successful tests already performed and the two three qualification campaigns already completed demonstrate the interest and robustness of the design solutions selected for the highly demanding operating conditions and confirm the thermo-structural Nozzle Extension contribution to the overall performance of the VINCI engine.

Topic: 14 - CMC Applications in Space Transportation
Abstract no. 2138

# Development of a cost-effective CMC for the manufacturing of the reusable Space Rider Body Flap Assembly

Presenting author: Dr CAVALLI Lorenzo
Affiliation: Petroceramics spa
Viale Europa 2,24040 Stezzano (BG) (Italy)
Co-authors: VALLE Massimiliano (Petroceramics spa), DE STEFANO FUMO Mario (CIRA - Italian Aerospace Research Center), RUFOLO Giuseppe (CIRA - Italian Aerospace Research Center), GARDI Roberto (CIRA - Italian Aerospace Research Center)

CMCs exhibit attractive properties for several engineering applications, especially where lightweight, high mechanical and high temperature stability are required. In particular CMCs favorably combine the lightweight and the toughness of polymeric matrix composites with the high temperature resistance and the hardness of ceramics. These features are very attractive for several aerospace applications, such as reentry vehicles, where materials face severe thermal shocks and plasma oxygen exposition.
Here is presented the development and manufacturing of the body flap assembly of the ESA Space Rider, a space transportation system for routine access and return from low orbit.
The body flap consists of a Cf-SiC composite obtained by liquid silicon infiltration (LSI). This process offers several advantages over other CMCs manufacturing techniques. It is relatively cheap and fast and doesn't require handling of dangerous gas. Moreover, it is suitable for manufacturing parts with complex shapes.
The body flap was produced starting from a phenolic-based pre-preg. The green part was shaped using a vacuum bagging technique. The body flap bearing attachments were manufactured using an innovative shaping technique specifically tailored to allow a fine control over the positioning of the Cf. Our newly-developed technology maximizes the mechanical strength of the obtained component.
Moreover an innovative and cost-effective coating deposition technique was developed. The coating efficiently protects the carbon fibers from thermal oxidation. Such technique allows to coat the CMC surface with silicon carbide, formed in-situ by the reaction between superficial free carbon and silicon vapors. This treatment leads to a solid, compact carbide layer showing an unprecedented degree of adhesion to the CMC surface.
Material reusability was demonstrated in the SCIROCCO test facility in which six orbital missions were simulated. The full scale body flap will be tested in flight-representative conditions in order to demonstrate its thermo-structural resistance.

Topic: 14 - CMC Applications in Space Transportation
Abstract no. 2141

# Black Engine CMC Space Propulsion Technology

Presenting author: M ORTELT Markus
Affiliation: German Aerospace Center DLR
Pfaffenwaldring 38 - 40,D-70569 Stuttgart (Germany)

Co-authors:

Today the field of space transportation becomes of higher interest, considering the growing competition world-wide. DLR follows with a new ceramic rocket engine concept the goal of increase of efficiency and reliability. On the other hand weight, cost and operational risk shall be reduced aiming on world-wide competitiveness in the future.
Concerning these requirements a specific rocket thrust chamber design, based primarily on the application of transpiration cooled porous and thermo-chemically resistant CMCs as inner combustion chamber liner material, is favored, aiming on the improvement of today's high performance standards, e.g. typical high performance main stage or upper stage propulsion systems.
After meanwhile more than twenty years of intensive technology development, DLR offers within its Black Engine technology program a large portfolio of fiber reinforced structure systems for functional components in rocket thrust chambers or entire rocket engines respectively, using additionally CFRP for load carrying structure components or transpiration lubricated CMC journal bearings for lifetime increase of rocket turbo pumps.
One major focus lies on high ratio of thermal and mechanical load de-coupling capability, promising rocket engines with lifetime and maintenance standards like aviation engines, which will be a significant progress for very often re-used main stages in the near future.
The excellently working exclusively transpiration cooled ceramic rocket thrust chamber system, leads now to further system improvement. First evaluations are ongoing considering a new injection cooling method, where the porous CMC wall takes inherently both the function of cooling and injection. This new method is applied in a brand new design concept, reducing significantly typical pressure loss in the combustion chamber process and promising more than 5 % of overall engine efficiency increase. Not only the increase of efficiency is important but also the increase of reliability by applying higher blow rates for higher inner wall safety.

Topic: 14 - CMC Applications in Space Transportation
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# From IXV to Space Rider : CMC Thermal Protection System evolutions

Presenting author: M VALVERDE Jacques
Affiliation: ArianeGroup
Rue de Touban,33185 Le Haillan (France)
Co-authors: PICHON Thierry (ArianeGroup), BERNARD Romain (ArianeGroup)

Space Rider is an ESA program with the objective of providing a reusable unmanned space laboratory to be operated up to two months in orbit. This vehicle will be developed on the basis of the Intermediate experimental Vehicle (IXV) atmospheric re-entry demonstrator which successful flight took place on February 2015.
Within the scope of this program, ArianeGroup is in charge of the Thermal Protection Structure (TPS) of the windward, hinge and nose assemblies of the vehicle. This development relies on the windward and nose assemblies TPS previously developed, tested, justified, produced and integrated on IXV. Ariane Group used a SepcarbInox® Ceramic Matrix Composite (CMC) protection system to provide a high temperature resistant non ablative outer mould line (OML) for enhanced aerodynamic control.
After a reminder of the IXV concept and mission, this paper describes the main requirements and new challenges to develop the Space Rider TPS in comparison with those of the IXV (new aerothermodynamic loads, long orbit life, landing concerns, reusability and refurbishment).

Topic: 14 - CMC Applications in Space Transportation
Abstract no. 1895

# Characterization and modeling of bending properties of continuous fiber reinforced C/C-SiC sandwich structure


Presenting author: Dr SHI Yuan
Affiliation: German Aerospace Center, Institute of Structures and Design
Pfaffenwaldring 38-40,70569 Stuttgart (Germany)
Co-authors: HEIDENREICH Bernhard (German Aerospace Center Institute of Structures and Design), KOCH Dietmar (German Aerospace Center Institute of Structures and Design)



Due to their excellent stiffness-to-weight ratio, sandwich structures are considered as typical lightweight structure and have been widely used in aerospace and construction industries. Compared to traditional sandwich structures made of cardboard, aluminum and polymer material, sandwich structures based on ceramic matrix composites (CMCs) can achieve significantly higher service temperatures and longer lifetime.
In this study, the sandwich structures based on continuous fiber reinforced cores and skin panels have been developed via Liquid Silicon Infiltration (LSI) and in-situ joining methods. For FE-simulation (Finite Element simulation), a realistic model was developed, and the mechanical performance of C/C-SiC sandwich structures under four-point-bending was analyzed. Bending and shear stiffness of the sandwich structures were determined by FE-simulation and analytical methods. The results obtained by modeling approaches were compared with experimental results and the comparison showed good correlation. The analytical and FE-simulation approaches have been further used to study the parameter effects and the influence of the different core structures on the bending properties respectively. The proposed different approaches are suitable to determine and simulate the mechanical properties of C/C-SiC sandwich structures and are applicable for further product development.

Topic: 14 - CMC Applications in Space Transportation
Abstract no. 2023

# Ablation behavior of C/SiC in wind tunnel

Presenting author: Prof WANG Yiguang
Affiliation: Beijing Institute of Technology
Zhongguancun South Road 5#,100081 Beijing (China)

Co-authors:

The ablation behavior is crucial to the application of carbon fiber reinforced silicon carbide (C/SiC) composites in thermal protection systems of spacecraft to enter the atmosphere or to cruise hypersonically in near space. Plasma wind tunnel is a suitable tool to simulate the service conditions of space vehicles. In this study, we systematically studied the ablation behaviors of C/SiC composites in plasma wind tunnel. The oxidation behavior of C/SiC composites in plasma is discussed. The results indicated that the oxidation under atomic oxygen condition dominated at low heat flux and stagnation pressure; however, rapid recession mechanism by sublimation and decomposition of SiC was observed at high heat
flux and stagnation pressure. The temperature jump phenomenon during the ablation performed under high heat flux and stagnation pressure was attributed to the exposure of the carbon fibers to the plasma flow after consumption of the SiC coatings.

Topic: 14 - CMC Applications in Space Transportation
Abstract no. 2309

# Advanced High Temperature Ceramic Matrix Composites for Space- and Hypersonic-propulsion applications

Presenting author: M SCHMIDT-WIMMER Stephan
Affiliation: ArianeGroup GmbH
Robert-Koch-Strasse 1,82024 Taufkirchen (Germany)

Co-authors:

Cost reductions as well as increased performance are placed as key element of development and application of advanced materials these days also in space- or hypersonic propulsion applications.
During the past decade, ArianeGroup Company (formerly Astrium/ Airbus Defence and Space), together with various partners, worked intensively on developing ceramic matrix composite (CMC) components for hypersonic engines, liquid rocket- as well as orbital propulsion systems and additional space-relevant equipment . The advantages for the developers are obvious–the low specific weight, the high specific strength over a large temperature range, and their great damage tolerance compared with monolithic ceramics make this material class extremely interesting as a construction material for above mentioned applications.
Besides material and process development in the field of CMCs itself, additionally the development of special metal/ceramic and ceramic/ceramic joining techniques was and is still investigated. Besides this the studying of nondestructive and moreover destructive testing in relevant environment is under examination for the purpose of testing, validation and qualification of such components.
This paper will give an overview about CMC technologies within ArianeGroup Germany with focus on the manufacturing-processes as well as the test techniques applied. Finally it will give a detailed outline of the future CMC development and manufacturing envisaged.

Topic: 14 - CMC Applications in Space Transportation
Abstract no. 1969

# Fine Structure Carbon-Carbon For Application In Satellite Antenna Reflectors

Presenting author: M ELAKOV Aleksandr
Affiliation: JSC Kompozit
4 Pionerskaya,141070 Korolev (Moscow region, Russia)
Co-authors: BOGACHEV Eugeniy (JSC Kompozit)

The substrate material for the reflector of the satellite antenna should fit a number of specific requirements: low density, high specific stiffness, very low coefficient of thermal expansion over a wide interval of temperatures, long term stability in space environments and high thermal conductivity in the direction perpendicular to the reflecting surface. Conventional carbon-carbon meets most requirements placed upon the antenna reflector materials except for surface roughness due to the fact that carbon fiber preforms structure inhomogeneity. The talk presents the properties of a new organomorphic carbon-carbon based on Ipresskon® non-woven reinforcing network. The preform has homogeneous fine structure (pore diameters range from 10 ?m to 60 ?m). Use of the carbon-carbon for the reflector antenna manufacturing allows to minimize the roughness of its surface up to Ra 0.10-0.15 at a coefficient of thermal expansion around 0.5·10-6 K-1. The characteristics of the reflector made from the CMC are demonstrated.

Topic: 14 - CMC Applications in Space Transportation
Abstract no. 2304

# Manufacture and testing of a novel hybrid nozzle assembly based on liquid silicon infiltration


Presenting author: Prof KOCH Dietmar
Affiliation: German Aerospace Center, Institute of Structures and Design
Pfaffenwaldring 38-40,70569 Stuttgart (Germany)

Co-authors: MAINZER Bernd (German Aerospace Center Institute of Structures and Design),
JEMMALI Raouf (German Aerospace Center Institute of Structures and Design),
KOCH Dietmar (German Aerospace Center Institute of Structures and Design)



The increasing exploration and use of space and the associated costs require not only the optimization of the launch masses of rockets but also an increase in the specific performance of the engine. Fiber-reinforced ceramic matrix composite structures, which can be used in rocket nozzles, connect these two components. By reducing the mass of the engine, an increase in performance and payload capacity can be achieved.
The development of a novel hybrid design of a nozzle assembly consisting of a fiber reinforced ceramic (CMC) part and an integrated fiber reinforced polymer (CFRP) outer shell is presented. The nozzle is manufactured via wet filament winding with the ceramic part being produced by additional pyrolysis and liquid silicon infiltration (LSI) process. The hybrid nozzle was successfully tested including the qualification test on the European Sounding Rocket Range (ESRANGE). It could be proved that the ceramic part of the nozzle shows the advantages of the high mass specific characteristic values and a sufficient high temperature resistance under extreme conditions while the CFRP outer shell performs as an ablatively cooled system.

Topic: 14 - CMC Applications in Space Transportation
Abstract no. 2271

# CMC Sandwich Development and Testing for High-Temperature Space Application


Presenting author: M REIMER Thomas
Affiliation: Deutsches Zentrum für Luft- und Raumfahrt (DLR)
Pfaffenwaldring 38-40,70569 Stuttgart (Germany)

Co-authors: HEIDENREICH Bernhard (Deutsches Zentrum für Luft- und Raumfahrt (DLR), KOCH Dietmar (Deutsches Zentrum für Luft- und Raumfahrt (DLR)



Sandwich structures have proved their potential in aerospace applications due to their excellent stiffness-to-weight ratio for a long time in structures manufactured as polymeric matrix composites. In order to make use of the high potential of sandwich structures also in high-temperature applications, where polymeric matrix composites or metallic designs cannot be applied, a structural sandwich design made from ceramic matrix composites (CMC) has been developed by DLR via applying the reactive melt infiltration technology, which has been in use for some time to produce C/C-SiC material. Both, the skins as well as the core of the sandwich, are fabricated from C/C-SiC. Currently, three core types are under development, honeycomb and grid-core as well as folded cores. In this work, the focus is on the thermal properties of C/C-SiC sandwich structures based on grid and folded cores with regard to the application in thermal protection systems and hot structures of re-entry spacecraft. High-temperature tests were conducted on sandwich samples of 100 x 100 mm which were heated from one side up to approximately 1000°C in steady-state. The temperature field on both sides of the sandwich on the face sheets was determined via thermocouples. The tests were done in vacuum and at specified pressures. Three variants of sandwich structures were tested: hollow grid-core, the hollow fold-core and a simplified variant with linear core elements filled with a ceramic-fibre insulation material. In addition, finite-element modelling was applied to simulate the tests and to determine apparent thermal conductivities for the composite structures. As a conclusion, an outlook is presented about the current steps in the development which aim at filling the more complex cores of the sandwich with insulating material in order to increase thermal insulation efficiency.

Topic: 14 - CMC Applications in Space Transportation
Abstract no. 2049

# Development of C/C-SiC regenerative combustor wall for scramjet engine with cooling channel


Presenting author: Dr KIM Seyoung
Affiliation: Korea Institute of Energy Research
152,Gajung-ro,34129 Daejeon (Daejeon, South Korea)

Co-authors: KIM Soo-hyun (Korea Institute of Energy Research), HAN In-sub (Korea Institute of Energy Research), SEONG Young-hoon (Korea Institute of Energy Research), BANG Hyung Joon (Korea Institute of Energy Research)



Hypersonic vehicles using a scramjet engine as a propulsion system have longer flight distance than a rocket propulsion system. In addition, it is attracting attention as a promising future aviation technology in the field of space launch vehicles, high-speed reconnaissance aircraft and transport aircraft as well as military weapons field. The purpose of this study is to develop the technology to cool the wall of the combustor of the scramjet engine by using the engine fuel to prevent deterioration of the combustion wall surface and to increase the efficiency by increasing the injection fuel temperature. For this purpose, carbon fiber reinforced ceramic composites with superior high temperature stability against metals were applied. The manufacturing process of the combustor wall and the forming the fuel flow channels inside the wall were studied. In addition, the combustor parts manufactured by LSI (Liquid Silicon Infiltration) method were applied to the actual scramjet combustor with the inlet flow velocity Mach 2.0 with 40bar fuel pressure to perform the actual environmental test. The temperature changes of CMC combustion wall surface by fuel flow was measured simultaneously with laser induced phosphor thermometry (LIPT) and 2-dimesional wall temperature map was obtained. After the actual application test, the regeneration cooling effect and the structural stability of CMC parts were also studied.

Topic: 14 - CMC Applications in Space Transportation
Abstract no. 2077

# Structural Analysis and Pressure Test for C/SiC Regenerative Cooled Hypersonic Combustor Panel


Presenting author: Dr KIM Soo-Hyun
Affiliation: Korea Institute of Energy Research
152, Gajeong-ro,34129 Dajeon (South Korea)

Co-authors: KIM Seyoung (Korea Institute of Energy Research), SEONG Younghoon (Korea Institute of Energy Research), BANG Hyung-Joon (Korea Institute of Energy Research)



Hypersonic scramjet engines are applied to space launch vehicles and high-speed transport aircraft as well as military weapons with a Mach 5 or higher speed. In this study, structural analysis and pressure test were performed to apply carbon fiber reinforced silicon carbide matrix composite (C/SiC) which is a high temperature heat resistant material, to regenerative cooling type combustor of scramjet engine exposed to over 2000K combustion environment.
Because the existing superalloys for high temperature are greatly degraded in physical properties over 1000K, we applied C/SiC composites with a heat resistance temperature of 1600K or more to the combustor rear side panel exposed to the hottest environment among the supersonic combustor system.
To verify the structural safety of C/SiC composite combustor panel with regenerative cooling channel, a 3D FE model for combustor panel was constructed and structural analysis was performed. The case study was carried out by using the parameters of combustor wall thickness, the shape of the flow path, and the shape of the reservoir. Since the pressure of the fuel in the regenerative cooling channel is expected to be about 40 bar maximum, a design pressure of 100 bar is set considering the safety factor of 2.5.
The C/SiC composite combustor panel was fabricated through the structural analysis and design, and the water pressure test equipment was constructed to confirm the failure characteristics and leakage. The pressure was gradually increased to 100 bar or more during the pressure test which verified the structural safety of the composite combustor panel with regenerative cooling channel.

# Topic: 15

# CMC Applications
# in Terrestrial Transportation
# and Industrial Systems

Topic: 15 - CMC Applications in Terrestrial Transportation and Industrial Systems
Abstract no. 2037

# Carbon Ceramic Brake Systems – outlook after 20 years of success

Presenting author: M ORLANDI Marco
Affiliation: Brembo SGL Carbon Ceramic Brakes
viale Europa 2,24040 Stezzano (Italy, Italy)
Co-authors: ORLANDI Marco (BSCCB)

Compositionally graded fibers-reinforced carbon (C) and silicon carbide (SiC) composites - also known as Ceramic Matrix Composites (CMC) - have rapidly become some of the most exploited materials in the advanced ceramics industry for high temperature demanding applications. Engineered CMCs are becoming more and more used in several strategic sectors such as transports, energy generation, tooling machines, personal protective equipment. Their lightweight, high thermal stability and functional properties such as low thermal expansion and good tribological behaviour play an increasing importance for several new commercial applications.
CMC brakes have been introduced for top class vehicles since the beginning of this millennium. After 20 years of success in the use of such high performance brake system, a review of the production process will be presented, starting from the design of the product till the characterization of the final product.
The request of the new applications and the new challenges coming from the market, that are the drivers for the development, will be explored and critically analyzed as well, to draw the future of the CMC brakes.

# High performance carbon fiber reinforced ceramics composites for future brake system in Chinese high-speed railway

Presenting author: Prof XIAO Peng
Affiliation: Powder Metallurgy Research Institute, Central South University
Lushan south road 932,410083 Changsha (Hunan, China)

Co-authors: XIAO Peng (Powder Metallurgy Research Institute Central South University), LI Yang (Powder Metallurgy Research Institute Central South University), LI Zhuan (Powder Metallurgy Research Institute Central South University)

As the Chinese high speed trains rapidly developed in the past decades, their running speeds are now up to 350 km/h, and even as high as 400 km/h in the future. Undoubtedly, the faster running speeds raised new demand and higher challenge in the brake systems including the structural design and optimization, new brake materials, etc. Currently, aiming to enable a safe and reliable braking for the faster and faster high speed train with favorable weight reduction, the high performance brake materials with excellent thermal resistance, light weight, and reliable and stable brake properties in long term are be of urgent demand as replacements for the metal brake disk. Motivated by the above-mentioned aims, a carbon fiber perform reinforced dual matrix (carbon and SiC) composite (C/C-SiC) with favorable thermal, mechanical and frictional properties as well as a good industrialization prospect is finally developed by our team after more than 10 year's effort. In this work, a brief introduction for manufacture process, microstructures, general physical properties, and tribological behaviors under some typical conditions, for instance, simulated emergency brake of C/C-SiC for high speed train were involved. The previous investigations indicated that the light-weight C/C-SiC composites with good reliability in long term are capable to be a very promising candidate for brake system used in Chinese high speed train and subway.

Topic: 15 - CMC Applications in Terrestrial Transportation and Industrial Systems
Abstract no. 2139

# Lightweight Metal-Ceramic Hybrid Brake Disc for Electric-Powered Vehicles: Concept and Prototype


Presenting author: M BALZER Thorsten

Affiliation: Department of Ceramic Materials Engineering, University Bayreuth
Ludwig-Thoma-Str. 36b,95447 Bayreuth (Germany)

Co-authors: LANGHOF Nico (Department of Ceramic Materials Engineering University Bayreuth), KRENKEL Walter (Department of Ceramic Materials Engineering University Bayreuth)



Ceramic brake discs made out of short fibre reinforced C/SiC material show exceptional tribological properties and outstanding wear characteristics. However, the high price of these ceramic brake discs prohibit a broader use. Therefore, a new approach for a lightweight metal-ceramic hybrid brake disc, which consists of an aluminium support disc lined with short fibre reinforced C/SiC ceramic segments, is presented. Aluminium is used for the supporting body due to its low density, high thermal conductivity, corrosion resistance and low cost. The ceramic segments are used for the friction surface of the hybrid brake disc as a result of their favourable tribological and wear properties. An overview is given on the potential application areas and on the construction, manufacturing and testing of said hybrid brake disc.

A potential use case of a mid-class sedan with a mass of around 1.8 t and maximum travelling speeds of up to 200 km/h is taken as a basis for the construction of the hybrid brake disc. The dimensioning of the brake disc was conducted with the aid of thermal finite element analysis methods, so that the critical temperatures at the joints stay within predefined boundaries, which are determined by joining methods and material properties. Furthermore, different joining methods are examined and benchmarked in the light of the use case. Hence a Prototype brake disc was manufactured and tested on the dynamometer of the University of Bayreuth where different characteristic values, like wear, coefficient of friction and different temperatures were measured. The results were then compared to the results of standard commercially available brake discs, which were also measured on the dynamometer. In addition, the material properties of the used short fibre reinforced C/SiC were determined by three-point bending tests and microstructural analysis.

Topic: 15 - CMC Applications in Terrestrial Transportation and Industrial Systems
Abstract no. 2224

# thin walled and light weight all oxide ceramic matrix composites (OCMC) structures as substitute for sheet metals

Presenting author: M PRITZKOW Walter
Affiliation: Walter Pritzkow Spezialkeramik
Adam Opel Str. 6,70794 Filderstadt (Germany)

Co-authors:

The use of ceramic matrix composites often focusses on very high temperature application. However, all oxide ceramic matrix composites (OCMC) have a great potential also at moderate temperatures as they have a much better performance compared to metal parts even below 1000°C. Some application examples will be addressed in the presentation.
While at room temperature steel has a much higher tensile strength than OCMC, steel loses 90% of its tensile strength at 1000°C while OCMC keep about 90% of their room temperature strength. By replacing metal parts with OCMC parts a much better long-term stability can be obtained which helps to prolong maintenance intervals dramatically. Another issue is the low density of OCMC with 2.8 g/cm³ for OCMC compared to 7.8 g/cm³ of steel. When an OCMC structure for a flying vehicle is designed and manufactured with the same wall thickness the overall weight can be reduced to only 36% of the alternative steel structure. This results in a much better flying performance of the vehicle when OCMC can be used. Furthermore, thermal expansion of OCMC (8.5*10^-6 1/K) is only half of thermal expansion of steel (17*10^-6 1/K). When parts are designed accordingly the fiber reinforced ceramics show much better thermomechanical behavior as thermal stresses and deformation can be minimized and cracks can be prevented compared to steel components.
The above mentioned advantages of OCMC will be discussed along different thin walled, complex 3-D structures which are in operation today.

Topic: 15 - CMC Applications in Terrestrial Transportation and Industrial Systems
Abstract no. 2013

# Net shape CMC components produced by composite flow molding, pyrolysis and liquid silicon infiltration


Presenting author: M BIANCHI Giovanni
Affiliation: SUPSI-DTI-MEMTI
Via Cantonale 2C,Galleria 2,6928 Manno (Ticino, Switzerland)
Co-authors: VODERMAYER Albert (icotec AG), ORTONA Alberto (SUPSI-DTI-MEMTI)



The present processing route shows how to obtain net shape ceramic matrix composites (CMC) artefacts by pyrolysis and reactive silicon infiltration of green components obtained by composite flow moulding (CFM). Starting materials consist of IM carbon fiber impregnated in PEEK matrix. Despite component shape complexity, the obtained microstructure is characterized by uninterrupted fibres across the part since they can be produced to net shape, avoiding machining. In this work a net shape CMC screw was manufactured as a representative component. This new method enables to shape CMCs into complex geometries for many structural applications such as: bolts, nuts, rivets, springs and even turbine blades maintaining continuous fibres.

Topic: 15 - CMC Applications in Terrestrial Transportation and Industrial Systems
Abstract no. 2305

# Carbon fiber based ceramic brake materials - concepts and applications

Presenting author: Prof KOCH Dietmar
Affiliation: German Aerospace Center, Institute of Structures and Design
Pfaffenwaldring 38-40,70569 Stuttgart (Germany)

Co-authors: HEIDENREICH Bernhard (German Aerospace Center Institute of Structures and Design), SHI Yuan (German Aerospace Center Institute of Structures and Design), KLOPSCH Linda (German Aerospace Center Institute of Structures and Design), KESSEL Fiona (German Aerospace Center Institute of Structures and Design)

Carbon fiber reinforced ceramics manufactured via liquid silicon infiltration (LSI) are developed since the late 1980s. Carbon fibers are infiltrated with a polymer based matrix which will be converted to carbon during the pyrolysis process and then reacted to SiC by LSI. The resulting C/C-SiC composites show high abrasion resistance, sufficient heat capacity and high corrosion stability. These properties make the composites attractive for passenger car brake systems. Today, brake discs are manufactured in serial production very efficiently, being reinforced with short fibers and coated with a frictional SiC rich layer on the surface. Apart from this application, also other frictional applications have been realized as e.g. brake systems for high velocity elevators or brake discs for the Airbus A400M propeller brake system. A detailed overview of various C/C-SiC friction materials and the necessary microstructural design adapted to the individual frictional application will be presented. Additionally, new concepts for brake materials, focusing on enhanced preform technologies, like knitting or stitching, combined with short fiber reinforced frictional layers, will be shown and their potential will be discussed.

Topic: 15 - CMC Applications in Terrestrial Transportation and Industrial Systems
Abstract no. 2188

# High performance CMC friction materials for emergency brakes

Presenting author: Dr LANGHOF Nico
Affiliation: University of Bayreuth, Ceramic Materials Engineering
Ludwig-Thoma-Str. 36b,95447 Bayreuth (Bayern, Germany)

Co-authors: BALZER Thorsten (University of Bayreuth Ceramic Materials Engineering), KRENKEL Walter (University of Bayreuth Ceramic Materials Engineering)

There is an increasing demand for high performance friction materials for emergency brakes in industrial and automotive applications, e.g. within the upcoming e-mobility sector. For e-cars, an emergency brake based on friction is still required, but as small and as light as possible.
Materials for such friction applications demand the thermomechanical properties and the damage tolerance, which are known from Ceramic Matrix Composites.
Hence, within this study different friction material candidates were studied on a self-designed dynamometer. C/C-SiC, fabric and short-fiber reinforced, and state-of-the art LowMet material were chosen as pads, with the dimension of 30x30x10 $mm^3$ (length x width x thickness). A steel disc and a C/C-SiC ceramic brake disc (diameter 380 mm) were applied as the regarding counterparts. These materials results in several friction pairings, which were evaluated at a frictional energy of 0.6 MJ, by applying a fly wheel (800 kg; 100 $kgm^2$) and a sliding speed of 20 m/s, at a friction diameter of about 150 mm. This rotational energy is related to the kinetic energy of a moving passenger car (m = 1.5 t) which is slowed down from 100 km/h to zero in an emergency braking. Several braking events were conducted with very high braking pressures between 30 MPa and 60 MPa. After the determination of the coefficient of friction, the wear rate and the temperatures of the friction couples, their tribological behaviour were studied. Therefore, cross sections were prepared for optical and scanning electron microscopy investigations. Additionally, the frictional surface and the surface roughness were analysed in order to identify the occurring friction and wear mechanisms.
For the best friction couple an application related emergency brake scenario was selected, in order to visualize the potential of the development of new friction pairings for industrial and automotive emergency brakes.

Topic: 15 - CMC Applications in Terrestrial Transportation and Industrial Systems
Abstract no. 1834

# Effect of powdered h-BN as addition on the microstructure and properties of C/C composites fabricated by chemical vapor infiltration


Presenting author: Dr LI Yang

Affiliation: Powder Metallurgy Research Institute, Central South University
Lushan south road 932,410083 Changsha (Hunan, China)

Co-authors: XIAO Peng LI Zhuan LI Zhichao ZHANG Beng



In current work, the carbon fiber reinforced/carbon-boron nitride (C/C-BN) dual matrix composites were fabricated via adding hexagonal boron nitride (h-BN) powders into the needled carbon felt and subsequent chemical vapor infiltration (CVI) process.
The microstructures, mechanical properties and tribological properties of the C/C-BN composites were investigated. Results indicated that compared to pure C/C composites, the texture of PyC in C/C-BN composites is regenerative laminar (ReL) and the size of PyC is smaller, resulting in a better interfacial bonding between fiber and matrix. Moreover, significantly enhanced compressive strength with higher isotropic stress-strain response in both vertical and parallel loading directions were achieved in C/C-BN. As for the friction test mating with 30CrSiMoVA counterpart, the C/C-BN presented higher coefficient of friction with less linear and mass wear rates. Current investigation indicated that the C/C-BN would be a better friction materials than C/C composites.

# Topic: 16

# CMC Application in aeronautic engines

Topic: 16 - CMC Application in aeronautic engines
Abstract no. 2267

# Ceramic Matrix Composites Taking Flight at GE Aviation


Presenting author: M STEIBEL Jim
Affiliation: GE Aviation
One Neumann Way,45215 Cincinnati (Ohio, United States)

Co-authors: DIX Bria



GE Aviation has been at the forefront of developing advanced materials for turbine applications, including the recent certification of Ceramic Matrix Composites (CMC's) on the LEAP engine. Key aspects of CMC technology maturation and industrialization at GE Aviation will be discussed in this Paper. Major steps in our technology maturation journey will be discussed, including material and component testing challenges, lifing methods development, and the role of process modeling in manufacturing scale-up. Key industrialization aspects will also be reviewed, such as the need for data analytics, the approach to establishing a quality control strategy, and the resulting supply chain capability.

Topic: 16 - CMC Application in aeronautic engines
Abstract no. 2314

# Application of Ceramic Matrix Composites to Deliver Value

Presenting author: M LAZUR Andrew
Affiliation: Pratt & Whitney
400 East Main Street,06118 East Hartford (Connecticut, United States)

Co-authors:

The Pratt & Whitney PurePower® geared turbofan engine introduced dramatic improvements in propulsive efficiency and noise reduction. To continue improving the overall efficiency of our engines, further improvements to thermal efficiency are targeted. Ceramic matrix composites (CMCs) are one technology that offers significant promise in this area. The higher operating temperature of CMCs compared to current state of the art single crystal castings will enable the reduction or elimination of component cooling air and may also enable higher combustion temperatures thereby increasing thermal efficiency. While CMCs offer tremendous promise, the cost of CMCs is a significant challenge. Multiple facets of cost must be addressed to deliver value to our customers and enable broad implementation of CMC technology. P&W will leverage broad expertise in the field of CMCs to optimize material systems for cost and performance. Cost reduction efforts will leverage system-level engineering optimization, integrated computational materials engineering, automation and continuous improvement methods through data and data analytics to deliver the next generation of high efficiency turbine engines.

Topic: 16 - CMC Application in aeronautic engines
Abstract no. 2258

# Brief history of ceramic matrix composites and their possible future applications for aeroengines


Presenting author: M GOMEZ Philippe
Affiliation: DGA Techniques aéronautiques
47 rue saint Jean,BP 93123,31131 Balma (France)

Co-authors:



In the beginning of the 90's the ceramic matrix composites (CMC) have been considered for aeroengine applications as their operating temperatures increased, approaching limit of the superalloys. Today their maturity has been demonstrated through several technological development programmes (plugs, flaps, blades…), some of which resulted in industrial production series.

The aerostructure and engine manufacturers have worked together to set up a way to obtain the required certification from airworthiness authorities for CMC industrial parts, more difficult to obtain when the component is critical.

The demonstrators and parts now in service not only provide feedback and validation of the mechanical properties of the materials measured in our laboratories but they also make it possible to know the behaviour of these materials in real life.

The superalloys won a reprieve with the enhancement of the thermal barrier systems coupled with cooling. As the operating temperatures still increase, the need of new materials re-emerges for the next generation of aero engines.

This presentation ends with a review of the problems that will have to be tackled for the CMCs to get their entry ticket into the futures aero-engines.

Topic: 16 - CMC Application in aeronautic engines
Abstract no. 2240

# Property evaluation of $Al_2O_3/RE_3Al_5O_{12}$ (RE=Y, Er and Yb) directionally solidified eutectic ceramics for potential application in engine environment

Presenting author: Dr SUN Luchao
Affiliation: Institute of Metal Research, CAS
High performance Ceramics Division,72 Wenhua Road,110016 Shenyang (Liaoning, China)
Co-authors: WANG Jingyang (Institute of Metal Research Chinese Academy of Sciences)

Directionally solidified eutectic (DSE) $Al_2O_3/RE_3Al_5O_{12}$ ceramics have shown high stiffness retention and excellent creep resistance at high temperature. These DSE ceramics have potential applications as high temperature turbine blades in future aeronautical turbines or thermal power generation systems. In this work, highly-dense $Al_2O_3/RE_3Al_5O_{12}$ (RE=Y, Er and Lu) bulk eutectic ceramics were prepared by optical floating methods. We studied the feasibility of these $Al_2O_3/RE_3Al_5O_{12}$ eutectic composites as high-temperature materials exposed to hot combustion environment. The microstructures and mechanical properties are studied. Especially, their hot steam resistance at 1400 oC and molten CMAS (calcium-magnesium alumino-silicates) corrosion at 1500 oC are investigated. The results show that as-prepared $Al_2O_3/RE_3Al_5O_{12}$ ceramics possess enhanced hot steam and CMAS resistance at high temperatures compared to BSAS ($BaO$-$SrO$-$Al_2O_3$-$SiO_2$), which is the currently commercialized environmental barrier coating. The present study broadens the understanding of the capability of $Al_2O_3/RE_3Al_5O_{12}$ against extreme combustion environment.

Topic: 16 - CMC Application in aeronautic engines
Abstract no. 1823

# Testing - Key to a better understanding of CMC

Presenting author: Dr ALBERT Benedikt
Affiliation: MTU Aero Engines AG
Dachauer Strasse 665,80995 Munich (Germany)

Co-authors: SIKORSKI Siegfried (MTU Aero Engines AG), HAUG Simon (MTU Aero Engines AG), HOFMANN Severin (MTU Aero Engines AG)

CMC-materials are very promising candidates for high-temperature lightwight desgin of future aero engines. However, there are still a lot of questions to be answered since this class is rather new in contrast to HT-alloys. At MTU, we have intensified our activities in that field over the last decade. In the LPT, temperature range is interesting for both Ox/Ox and SiC/SiC-materials. Testing of CMC-materials is the base for benchmarking and understanding the behaviour in engine-relevant atmospheres. Valid test results are necessary for analytical assessment and modelling. This presentation shows some examples of tests conducted at MTU as well as some deductions taken from it. Main focal topics are: Combination of destructive and non-destructive testing, QA-aspects of testing as well as possibilities of generating as much information as possible from evaluated specimens and geometries.

Topic: 16 - CMC Application in aeronautic engines
Abstract no. 2276

# Advances in developing an alternative to ceramic matrix composites in high temperature applications

Presenting author: Prof MILEIKO Sergei
Affiliation: Institute of Solid State Physics of Russian Academy of Sciences
2 Ossipian str,142432 Chernogolovka (Russia)

Co-authors:

CMCs that were born in Bordeaux by Naslain's team are excellent high temperature materials for many applications. However, there are some structures that need to be made of materials with a metal-like behavior as they are experienced very complicated loadings in severe external conditions. An example of such structures is the turbine blade, temperature of which determines the thermal efficiency of gas turbines to a large degree.
The present author with his colleagues presented at HTCMC-8 results of experiments with oxide-fibre/molybdenum-matrix composites produced by the internal crystallization method (ICM). The realization of ICM includes preparing a molybdenum carcass composed of foil and wires to be then infiltrated with an oxide melt to produce the reinforcing oxide component. The carcass can be easily made of pure molybdenum and can hardly be made of modern molybdenum alloys containing high volume fractions of brittle silicide and T2-phase, those composites were characterized by not sufficiently high mechanical properties at high temperatures.
Hence, the author's team have developed a method to harden the molybdenum matrix by introducing sufficiently large volumes of ceramic particles (Mo3Si and Mo5SiB2) into the matrix on a stage of preparing the molybdenum carcass made of pure molybdenum. The method is based on the usage of a slurry containing silicon and boron.
Composites with various oxide fibres and Mo+Mo3Si+Mo5SiB2 matrix have strength characteristics higher than simple oxide/Mo composites (OMCs).
For evaluation of resistance to the corrosion in the propane-butane flame of a temperature up to about 1400oC the specimens were coated with yttrium molybdate and molybdenum disilicide layers of various thicknesses and microstructures. Preliminary test results did show sufficiently high stability of the composite microstructure.

Topic: 16 - CMC Application in aeronautic engines
Abstract no. 1774

# Development and evaluation of the fracture toughness of SiC-based ceramic matrix composite.

Presenting author: Mme DELAGE Justine
Affiliation: Imperial College London
85 Cobbold ROad,W12 9LA London (United Kingdom)

Co-authors:

Since the 1980s, fibre-reinforced ceramic matrix composites (CMCs) are the subject of extensive developments as candidate materials for structural applications in extreme environments, due to their excellent thermomechanical properties. A promising use of SiC/SiC CMCs would be the replacement of metallic super alloy components of aircraft engines. CMCs are less heavy and can be used at higher temperatures than metallic components. Thereby, their uses could increase the efficiency of the engine and reduce the consumption of fuel, leading to less pollution and economic gains.
Nonetheless, the main complication with CMCs is that they exhibit a complex fibre related crack propagation during fracture which makes their mechanical behaviour hard to fully understand and to model. More specifically, the measurement of the fracture toughness, which describes the resistance to crack propagation of a material, is particularly complicated because of the composite fracture mechanisms. These multiple mechanisms don't lead to a single straight crack initiating at the notch tip but to jagged macroscopic crack due to a combination of matrix microcracks, fibres bridging and pull-out mechanisms.
The present study focusses on the understanding of the fracture behaviour, and especially on measuring the fracture toughness of SiC/SiC CMC. To do that, tensile tests and bending tests have been carried out. Tensile tests give the opportunity to determine the resistance to crack propagation by notch sensitivity testing at ambient temperature in air atmospheric pressure. Bending tests allow us to observe in-situ the development of cracks within the composite microstructure and to identify and understand better the fracture mechanisms of the composite. Both tests may lead to the measurement of an equivalent fracture toughness of the composite.

Topic: 16 - CMC Application in aeronautic engines
Abstract no. 2289

# Advanced High Temperature Materials in Next Generation Vertical Lift Propulsion Systems


Presenting author: Dr GHOSHAL Anindya
Affiliation: CCDC Army Research L:aboratory
B4603, 6340 Rodman Road,21005 Aberdeen Proving Ground (maryland, United States)
Co-authors: MURUGAN Muthuvel (CCDC US Army Research Lab), WALOCK Michael (CCDC US Army Research Lab), BRAVO Luis (CCDC Army Research Lab), MOCK Clara (CCDC Army Research Lab), NIETO Andy (Naval Postgraduate School), PEPI Marc (CCDC Army Research Laboratory), HIRSCH Samuel (CCDC Army Research Lab), DOWDING Robert (CCDC Army Research Lab)



Aviation propulsion system structures are subjected to challenging conditions such as extremely high velocities, ultra-high/low temperatures, and excessive dust/sand/smoke/volcanic ash conditions during military operations. Therefore, the research and development of high-performance engine materials with superior characteristics such as great mechanical strength, high fatigue resistance and creep resistance, good tolerance to wide temperature variations, and excellent resistance to corrosion and oxidation is essential to the evolution of highly robust and efficient propulsion systems without performance degradation, even in hostile environments.

Next generation Army Brayton Cycle Engines require hot section components to survive higher temperature (>1550 deg C) and pressure (> 30 bar). Current generation of state of the art intermetallics and single crystal high temperature alloys with thin film cooling and thermal barrier coatings cannot survive the high temperature. The goal of this collaborative research program in advanced high temperature propulsion materials within United States Army Research Laboratory (ARL) is multifold: 1) CMC Material and Interface Characterization 2) Sandphobic T/EBC and Surface Modification of CMC 3) CMC Sensing and NDE and iv) Ultra-High Temperature Ceramic (UHTC) Composites.

ARL's proposed solution is to engineer advanced Ceramic Matrix Composites with Thermal/Environmental Barrier Coating and with and without film cooling to withstand relevant high temperature and pressure. It is a tiered approach: Near term approach is to develop a hybrid material system with T/EBC and CMC bulk substrate with thin film cooling as needed. Far term goal is to develop an integrated graded low-k high strain tolerant material system with CMAS-phobic and erosion resistant capabilities. The UHTC composites is one of the approaches for bulk materials under the integrated functionalized ceramics research. High Temperature Sensing on CMCs is another aspect of ARL research in this area. ARL with AMRDEC, NASA GRC and NAVAIR are leading the research into advanced high propulsion material systems.

Topic: 16 - CMC Application in aeronautic engines
Abstract no. 2260

# Advanced exhaust design based on new 2D oxide/oxide materials for civil aircraft engine


Presenting author: Dr BOUILLON Florent
Affiliation: Safran Ceramics
rue de Touban,33185 Le Haillan (France)

Co-authors: GUEL Nicolas (Matéis INSA Lyon / IRT Saint Exupéry), SALLY Orianne (LMT Cachan / ONERA / Safran Ceramics), BROUTELLE Marion (ISAE supaero / IRT Saint Exupéry), MALENFANT Jean-Charles (SAFRAN CERAMICS)



In order to meet performance and environmental challenges, SAFRAN is developing a new generation of exhaust structures based on an oxide-oxide composite. Oxide/oxide composites are considered as performant material for such parts subject to thermomechanical loading. They have the advantage of being physically and chemically stable over a wide temperature range, up to 800°C and to have an adequate mechanical behaviour.
The presentation will explain how, starting from the specificities of the part, and the constraints related to certification, the material developed by SAFRAN has been studied and then optimized in order to obtain an efficient structure. The analysis of the material under different loading conditions, using multi-instrumentation will be presented. Bearing behaviour of such material will be detail as design key point. A proposal of a new limit load criteria will be also done in accordance with CS 25 certification text.
The simulation of the mechanical behaviour and life duration prediction of these parts are still a major challenge for civil applications, where the number of flight cycles is important and safety requirements are high. The presentation will conclude with the progress made in the field of justification to support the certification of these structures. In particular, macroscopic approaches that can be used will be detailed.

Topic: 16 - CMC Application in aeronautic engines
Abstract no. 2299

# CMC FRACTURE TOUGHNESS TEST STANDARD DEVELOPMENT


Presenting author: Dr ABDI Frank
Affiliation: Chief Scientist
5150 E. Pacific Coast Highway,Suite # 650,90804 Long Beach (CA, United States)
Co-authors: BAID Harsh (AlphaSTAR Corporation), EFTEKHARIAN Amir-Hossein (AlphaSTAR Corporation), GODINES Cody (AlphaSTAR Corporation), MORSCHER Gregory (University of Akron), CHOI Sung (NAVAIR), SHI Jun (Rolls Royce Corporation), AHMED Jalees (AlphaSTAR Corporation), GONCZY Steve (Gateway Materials)



The objective of this effort is to develop and demonstrate ASTM standard interlaminar Mode I and Mode II fracture toughness analysis and NDE test methods for Ceramic Matrix Composites (CMC). Interlaminar Crack Growth Resistance (CGR) Mode I and Mode II tests may exhibit mixed mode interaction and zig-zag crack growth pattern, wavy cracks, and fiber bridging. The experimental parameters that may contribute to the difficulty are summarized as, specimen width and thickness, interface coating thickness, mixed mode failure evolution, and interlmainar defects. A CGR solution Strategy was developed and validated for SiC/SiC, and Oxide/Oxide CMC by four functional pillars interactively working together: 1) Advanced Multi-Scale Modeling and prediction of CGR; 2) Room/High temperature (RTD/ETD) tests with AE/ER (acoustic emission, and Electrical resistivity), and Digital Image Correlation (DIC) measurements to monitor and observe crack extension to assess the viability; 3) Interpretation of test and model results with analytical CGR and derivation of compliance equation for sharp, and blunt notch; and 4) ASTM ballot submittal Round Robin exercises. The optimized CGR Multi-Scale Progressive Failure Analysis (MS-PFA) predictive results was validated with standard test methods: a) Mode I Wedge loaded, double cantilever beam (DCB, WLDCB); and b) Mode II- End Notched Flexure (ENF) and End-Loaded Split (ELS) at RTD/ETD. ICME predictive methodology of Mode I-II Interlaminar CGR; and a new ASTM standard simplified CGR compliance equation was validated. Mode I was characterized where rising R-curve behaviors were observed signifying increased toughness/resistance to crack growth. DOE methods were used to optimize Mode I-II Specimen geometry and initial crack length while optimizing the contribution of ILT and ILS, Anisotropic stiffness matrix was measure by ER and predicted by MS-PFA within good accuracy.

Topic: 16 - CMC Application in aeronautic engines
Abstract no. 2110

# Fatigue Behavior of BN Particle Dispersion SiC Composites Fabricated with Prepreg Technique

Presenting author: Prof HINOKI Tatsuya
Affiliation: Kyoto University
Gokasho,611-0011 Uji (Kyoto, Japan)

Co-authors: KAWASAKI Kanjiro (Kyoto University), SHINODA Fujio (Kyoto University), TSURUI Nobuhito (Kobe Material Testing Laboratory Co. Ltd.), HOKARI Shota (Kobe Material Testing Laboratory Co. Ltd.), SHIMODA Kazuya (National Institute for Material Science)

Silicon carbide composites basically require weak fiber/matrix interphase like boron nitride (BN). However precise control is the critical issue in terms of large scale production and cost. To form complicated shape without debonding of the interphase is another significant issue. The interphase is the weakest link for the environmental effects. The SiC composites were developed by applying BN particle dispersion in SiC matrix without the interphase. The objective is to understand fatigue behavior of the SiC composites.
Silicon carbide composites reinforced with satin weave Hi-Nicalon type-S were fabricated by liquid phase sintering using the prepreg sheet. The prepreg sheet consisted with SiC fibers, SiC and BN powders and Al2O3 as sintering additive. Mechanical properties were characterized by tensile and fatigue tests.
The BN particle dispersion SiC composites had uniform microstructure through thickness. No significant degradation of strength wasn’t observed at 1200C in air. Oxidation of the composites were limited to near surface. The composites didn’t break following over one million cycles applying 175 MPa at 1200C in air. The specimens had no oxidation resistant coating like CVD SiC.
This work was supported by METI, Japan for the Advancement of Strategic Core Technologies, “Development of Production Process of SiC/SiC composites for High Efficiency Aircraft Engine.”

Topic: 16 - CMC Application in aeronautic engines
Abstract no. 1990

# SiC-based Ceramic Matrix Composite behavior enhancement for Gaz Turbines Hot Sections

Presenting author: M BOUILLON Eric
Affiliation: Safran Ceramics
Safran Ceramics,Rue de Touban,33185 Le Haillan (33, France - TOM)

Co-authors: LACOMBE Benjamin (Safran Ceramics), RICHARD Yann (Safran Ceramics)

Silicon carbide fibers reinforced silicon carbide based-matrix composites (SiC/SiC CMC's), are probably becoming a major leading alternative for the design and manufacturing of the next gaz turbine engines hot parts as airfoils, shroud, combustion chambers. These materials offer higher temperature capability than the current state-of-the-art metallic superalloys. The growing interest in CMC technologies development is directly linked to the new short-term engine design constraints in the context of air travel booming, namely : a drastic decreasing of polluting emissions and a specific fuel consumption decrease.
Building on past materials efforts in the field of both space launchers and military engines aircraft, Safran continued to enhance CMC technologies for commercial aircraft engines. Considering this new target, one of the key issues related to this emerging technology is to develop and industrialize materials offering high thermomechanical design allowables and stable lifetime properties, in representative environment. To reach these goals, an important work has been done, in the implementation of Melt –Infiltrated SiC/SiC CMC.
In order to limit the risk of material damage under operating conditions, a design criteria below the elastic limit must be applied. For SiC/SiC CMC, this level is driven by the Matrix Cracking Strength (MSC) limit. Therefore, an iterative approach based on an optimization of each block, coupled with precise mechanical behavior studies, at meso and macro scale, was carried out. This allowed us to define a robust manufacturing process, leading to a MSC of around to 250MPa.
The CMC technology development also requires an extensive work of thermomechanical damage behavior analysis, based on components tests, in increasingly representative conditions. In this presentation, a brief description of the CMC enhancement approach is proposed, including material allowables proposal and component behavior analysis.

Topic: 16 - CMC Application in aeronautic engines
Abstract no. 1828

# Development and evaluation of sub-element testing of SiC/SiC ceramic matrix composites at elevated temperatures

Presenting author: Mme GALE Louise
Affiliation: Rolls-Royce Plc
23 Springfield Road,Chellaston,De73 5sl Derby (United Kingdom)
Co-authors: HARRIS Stephen (Rolls-Royce Plc), PATTISON Stephen (Rolls-Royce Plc), REIS Fabio (Rolls-Royce Plc)

SiC/SiC ceramic matrix composites (CMCs) are being developed for use in aero-engines to replace nickel superalloy components. CMCs offer reduced weight and higher temperature capability which will enable higher efficiency and lower emission aero-engines. CMC components will have complex geometries and be subjected to mixed-mode stress states which will change through a flight cycle. Hence mixed-mode mechanical tests, such as sub-elements, are required to better understand the effect of these stress states on damage progression and failure within the CMC.
This study will present the development of high temperature C-shape sub-element testing with the use of Digital image correlation (DIC) to indicate the spatial distribution of strain accumulation and subsequent development of cracking. Test data will be presented from monotonic and fatigue tests and compared with predicted behaviour from modelling. Fractographic assessment of tested specimens will be used to study in-plane and interlaminar damage mechanisms and how they interact. Finally sub-element and flexural testing will be compared to study the effect of geometry on damage progression in mixed-mode mechanical stress states.

Topic: 16 - CMC Application in aeronautic engines
Abstract no. 1926

# Microstructure and Properties of C/SiC Composites Prepared by Reactive Melt Infiltration

Presenting author: Dr WANG Peng
Affiliation: Research Institute of Aerospace Special Materials and Processing Technology
Fengtai District Yun Gang Bei Li 40,100074 Beijing (Beijing, China)

Co-authors:

C/SiC ceramic matrix composites are possession of high temperature resistance and high specific strength and have been widely used in aerospace propulsion. C/SiC composites were prepared by reactive melt infiltration (RMI) of molten Si into the C/C porous preform. The process mainly consists of pyrolyzing C fiber preform which has been impregnated by resin into a porous C/C preform under high temperature, and then impregnating melted silicon at a higher temperature to form SiC matrix. Therefore, determining the mechanism of resin pyrolysis?controlling the liquid silicon infiltration and reaction process is of great significance for controlling the microstructure and mechanical properties of C/SiC composites. In this paper, the mechanism of furan resin pyrolysis is inferred by analyzing the gas products in the process of pyrolyzing. The obtained C/C porous preform was then subjected to high temperature treatment to study the effect of pore structure and graphitization degree on the silicon infiltration process. Influences of C/C porous preform embedded in silicon powder at different positons on the capillary absorption behaviors were investigated. Lots of free Si can be found inside of C/SiC composites prepared by RMI. The free Si was decreased significantly after desilication treatment of C/SiC composite, the composite density and flexural strength decreased as well.

Topic: 16 - CMC Application in aeronautic engines
Abstract no. 1320

# Micro-Mechanical Testing of the BN Interlayer in SiCf/SiC Composites for Aero-Propulsion

Presenting author: M DE MEYERE Robin
Affiliation: University of Oxford
St Edmund Hall,Queen's Lane,OX1 4AR Oxford (Oxfordshire, United Kingdom)
Co-authors: ARMSTRONG David E.J. (University of Oxford), MARROW T. James (University of Oxford)

Ceramic Matrix Composites (CMC's) are finding renewed interest in the aerospace community for use as high temperature components in engines due to the potential for cooling air reductions over metallic parts, amongst other benefits such as weight saving and improving the turbine blade clearance. Quasi-brittle SiCf/SiC composites are toughened by the application of a boron nitride interphase coating to the fibre, which allows for cracks to deviate from the matrix. The principal issues faced by SiC-based composites lie in their degradation in corrosive environments (changing the interphase region and embrittling the overall composite) and their current inadequacy to adopt performance life models. Therefore, maintaining the interfacial properties of the composite at high temperatures is crucial. The extraction of these said properties has however proven itself to be a major engineering challenge in materials science. A few meso-scale and macro-scale techniques such as the transverse bend test and the Brazilian disc compression test have shown experimental reproducibility but are unsupported by sufficient modelling. The most accurate method for determining the properties at the micro-scale remains the push-out method on singular fibres. Herein the talk will present current both advances in using the fibre push-out method and some of the challenges to overcome with push-outs in order to accurately measure the interfacial shear stresses, coefficients of friction and residual compressive stresses at the fibre/matrix interface. The push-out method will be contrasted to the fibre push-back and push-in techniques and a novel 'via' push-out method will be introduced. Finally, suggestions for improving the method to corroborate with ongoing modelling work will be showcased.

# Topic: 17

# Advanced materials for sustainable energy (incl. nuclear fission and fusion, industrial gas turbines)

Topic: 17 - Advanced materials for sustainable energy (incl. nuclear fission and fusion, industrial gas turbines)
Abstract no. 2365

# Advanced Manufacturing of Ceramic Materials and Components for Energy Systems


Presenting author: Dr TERRANI Kurt
Affiliation: Oak Ridge National Laboratory
1 Bethel Valley Rd,37831 Oak Ridge (TN, United States)

Co-authors:



Application of ceramics into energy systems allows for major improvements in performance parameters of those systems. In general, superior thermophysical, environmental, and mechanical stability of ceramics over their metallic counterparts allows for higher temperatures that in turn facilitate improved energy system efficiency. However, conventional ceramic manufacturing routes offer limited options for realizing complex geometries that are characteristic of functional systems. Advanced manufacturing allows for realizing high-quality ceramic components with complex geometries that are otherwise simply not viable. Examples of advanced manufacturing techniques applied to refractory carbides are discussed in detail. Performance characteristics of resulting components are summarized. Finally, and of great importance, application of advanced manufacturing process monitoring to allow for prediction of final component quality is also discussed.

Topic: 17 - Advanced materials for sustainable energy (incl. nuclear fission and fusion, industrial gas turbines)
Abstract no. 1821

# Oxidation of SiCf-SiC CMC cladding tubes for GFR application in impure helium atmosphere up to 1600°C


Presenting author: Dr STEINBRüCK Martin
Affiliation: Karlsruhe Institute of Technology (KIT)
Hermann-von-Helmholtz-Platz 1,76344 Eggenstein-Leopoldshafen (Deutschland, Germany)
Co-authors:



This paper presents results obtained in the framework of the European MatISSE program on materials innovation for a safe and sustainable nuclear power generation.
The oxidation kinetics of SiCf-SiC cladding tube segments in impure helium atmosphere prototypical for GFRs was investigated at ambient pressure in the temperature range between 900°C and 1600°C using a thermo-gravimetric device. The transition from passive oxidation (formation of a protective silica scale) to active oxidation (volatilization of silica due to the formation of SiO and other volatile species) occurred between 1200°C and 1300°C. Hence, during target operational temperatures for a GFR of 900-1000°C, the CMC cladding material should withstand long-term operation with respect to oxidation and corrosion. At higher temperatures beyond 1300°C, i.e. under accident conditions, volatilization of composite constituents would lead to serious degradation of the SiC-based cladding tubes.
In addition, the interaction between the tantalum layer and silicon carbide as applied in so-called sandwich tubes was investigated.

Topic: 17 - Advanced materials for sustainable energy (incl. nuclear fission and fusion, industrial gas turbines)
Abstract no. 2291

# Accident-tolerant SiC/SiC composite fuel cladding materials – the H2020 IL TROVATORE approach to performance optimization (keynote)

Presenting author: Prof LAMBRINOU Konstantina
Affiliation: SCK-CEN
Boeretang 200,2400 Mol (Belgium)

Co-authors:

The 2011 Fukushima Daiichi event demonstrated the need for improved nuclear energy safety, thus fully justifying the global R&D activities on the development of accident-tolerant fuels (ATFs). The main objective of the H2020 project IL TROVATORE is the performance optimization of select ATF cladding material concepts, followed by their validation in an industrially-relevant environment by neutron irradiation in PWR-like water. The IL TROVATORE candidate ATF clad concepts comprise SiC/SiC composites, coated and surface-modified clads, and ODS-FeCrAl alloys. The SiC/SiC composite clads, in particular, enjoy major industrial investments in Europe, the USA and Japan, due to the recognized potential of SiC for true accident tolerance in beyond-design-basis accidents (>1200 degC). However, all technical challenges associated with the use of SiC/SiC composites have not yet been addressed, making their qualification and licensing time-consuming and costly. Two known challenges regarding the use of SiC/SiC materials in LWRs revolve around the identification of reliable tube sealing approaches, and the appreciable material loss rates in water (nominal operation), due to the silica formation and dissolution. A newly identified problem that endangers the high-temperature accidence tolerance of SiC/SiC composites, limiting it to 1705 degC (beta-cristobalite melting point), is caused by the melting of silica that forms on the SiC surface in steam (transients/accidents). Molten silica reacts with SiC, producing gaseous species (SiO, CO), and compromising the SiC/SiC integrity. This lecture presents the challenges in performance optimization of SiC/SiC composite clads, starting from material production and joining, to coolant (water, steam) compatibility, and radiation tolerance. Moreover, it considers methods that prevent the in-service formation of silica, thus exploiting the SiC refractoriness (melting point 2830 degC), and assesses the radiation tolerance of various SiC/SiC composites using ion/proton irradiation.

Topic: 17 - Advanced materials for sustainable energy (incl. nuclear fission and fusion, industrial gas turbines)
Abstract no. 2143

# Opportunities for High Temperature Ceramics and Composites in Fusion Energy Applications


Presenting author: Dr KATOH Yutai
Affiliation: Oak Ridge National Laboratory
1 Bethel Valley Rd,37831 Oak Ridge (Tennessee, United States)

Co-authors: KOYANAGI Takaaki (Oak Ridge National Laboratory), BHATTACHARYA Arunodaya (Oak Ridge National Laboratory), PARISH Chad (Oak Ridge National Laboratory), EDMONDSON Philip (Oak Ridge National Laboratory), ANG Caen (University of Tennessee Knoxville), SNEAD Lance (Stony Brook University)



A long-standing challenge for the successful development of fusion energy involves development of high-performance blanket and plasma-facing component materials that withstand the harsh operating environment of fusion power reactors. While the current mainline fusion materials such as the reduced-activation ferritic/martensitic (RAFM) steels and tungsten are considered promising for blanket structures and plasma-facing surfaces, respectively, novel high temperature materials present the potential to enable break-through concepts for those components. Examples of novel material classes of interest include ultra-high temperature ceramics (mainly carbides and borides) that are significantly more refractory than metallic refractories, and so-called MAX-phase ternaries which may present acceptable radiation tolerance. This paper discusses these novel/emerging high temperature ceramics and their composites for fusion energy applications for their advantages, promises, and challenges.


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Topic: 17 - Advanced materials for sustainable energy (incl. nuclear fission and fusion, industrial gas turbines)
Abstract no. 1858

# Enhancing accident tolerance of nuclear fuel with SiC-based cladding


Presenting author: M LORRETTE Christophe
Affiliation: CEA
Nuclear center of Saclay,91191 Gif-sur-Yvette (France)

Co-authors: BRAUN James (DEN-Service de Recherches Métallurgiques Appliquées CEA Université Paris-Saclay), SAUDER Cédric (DEN-Service de Recherches Métallurgiques Appliquées CEA Université Paris-Saclay), PALANCHER Hervé (DEN-Département d'Etude du Combustible CEA), DUQESNE Loys (FRAMATOME Fuel), POUILLIER Edouard (EDF R&D)



Following Fukushima-Daiichi nuclear disaster, French Nuclear Institute was led to consider silicon carbide based continuous fiber ceramic matrix composite materials (SiCf/SiC) as a long-term option for Gen III/III+ light water reactor (LWR) cladding to enhance the accident tolerance of the fuel (ATF). In this respect, the extensive R&D activities have resulted in significant progress in the fabrication of representative and functional ceramic specimens, removing some technological barriers preventing use in a nuclear environment. In addition to on-going research, a collaborative program assessed the thermo-mechanical performances of SiCf/SiC composites produced at CEA and collected the required data for conceptual design.
The presentation will give an overview of the on-going developments and progress, including the proven benefits expected from a SiC-based fuel cladding concept as ATF solutions. The technical challenges to overcome and data gaps to fill before commercial deployment will be also highlighted.

Topic: 17 - Advanced materials for sustainable energy (incl. nuclear fission and fusion, industrial gas turbines)
Abstract no. 2223

# Industrial manufacturing of a novel graphite-metal carbide composite with potential application under extreme environments


Presenting author: Dr RIVERA MONTE Sergio
Affiliation: Nanoker Research S.L.
Polígono Olloniego Parc 22A Nave 5,33660 Oviedo (Asturias, Spain)

Co-authors: GUTIERREZ Carlos (Nanoker Research S.L.), FERNANDEZ Adolfo (CINN-CSIC), TORRECILLAS Ramón (CINN-CSIC)



The use of graphite as a structural material for extreme applications has been always limited because of its intrinsic low mechanical properties (i.e. maximum flexural strength of around 60 MPa). On the other hand, metal carbides have only limited applications as high temperature refractory materials due to poor thermal shock resistance and problems during manufacturing.
The graphite-metal carbide composites are promising materials to be used in structural applications under extreme environments as they can keep several characteristic properties of graphite (low density, high thermal shock resistance, high electrical conductivity, high thermal conductivity) whilst their mechanical properties increase thanks to a reinforcement of the metal carbide and a reduction of the total porosity of the material.
In this work, we develop a novel graphite-metal carbide composite by the spark plasma sintering (SPS) technology up to an industrial blank size with a diameter of 175 mm and a thickness of 30 mm. This material was invented by CERN and Brevetti Bizz, through support from the ARIES H2020 project and from the High Luminosity LHC (HL-LHC) project. Nanoker has upscaled the material manufacturing and has been awarded with a tender from CERN to manufacture up to 380 blocks in this material that will be installed in the collimator jaws in the next LHC upgrade.
This material has resulted in an outstanding combination of properties with a strength higher than 100 MPa, an electrical conductivity in plane of around 700 W/m·K, a thermal expansion coefficient lower than 2·10-6ºC-1 and a final density of 2.6 g/cm3. The material is envisaged for those applications where a great amount of heat dissipation is required as well as where graphite is limited due to its low mechanical properties as this composite almost double its performance.

Topic: 17 - Advanced materials for sustainable energy (incl. nuclear fission and fusion, industrial gas turbines)
Abstract no. 2042

# TiNb2O7/keratin-derived carbon microtubes composites as anode materials for lithium-ion batteries


Presenting author: Dr T Ganesh Babu
Affiliation: Indian Institute of Technology Madras
Department of MME,IITM,600036 Chennai (Tamilnadu, India)

Co-authors: SHANMUGAM Vasu (Centre for Automotive Energy Materials International Advanced Research Centre for Powder Metallurgy and New Materials IITM Research Park Kanagam Taramani Chennai 600113 India), A Noel (Laboratory for High Performance Ceramics Department of Metallurgical and Materials Engineering Indian Institute of Technology-Madras (IIT Madras), Chennai 600036 India), MOODAKARE Sahana B. (Centre for Automotive Energy Materials International Advanced Research Centre for Powder Metallurgy and New Materials IITM Research Park Kanagam Taramani Chennai 600113 India), N V Ravi Kumar (Laboratory for High Performance Ceramics Department of Metallurgical and Materials Engineering Indian Institute of Technology-Madras (IIT Madras), Chennai 600036 India)



TiNb2O7 (TNO) is a promising intercalation type anode material for lithium-ion batteries (LIBs) owing to its high theoretical capacity (~388 mAhg-1). However, the practical application of TNO is limited by its poor rate capability resulting from low electronic conductivity and poor ionic diffusivity which needs to be urgently addressed to capitalize the intrinsic advantage of this class of anode materials. In this regard, porous-TNO and its keratin-derived carbon microtubes (TNO/CMT) composites were prepared by sol-gel method followed by pyrolysis under nitrogen atmosphere to yield high surface area and conductive functional materials. With pristine TNO, TiNb2O7 crystalline phase is formed with specific surface area (SSA) of 28 m2g-1, whereas TNO/CMT yields TiNb2O7 and non-stoichiometric (Ti0.712Nb0.288) O2 as crystalline phases with SSA of 89 m2g-1. Morphological analyses through HRTEM revealed existence of good contact between the CMT and the TNO nano-crystallites which assisted in reduction of crystallite size and improvement in electrical conduction. The formation of reduced phase along with reduction in crystallite size in the case of TNO/CMT resulted in better electrochemical performance in contrast to pristine TNO. TNO/CMT also exhibited significantly better rate capability and cyclic stability than TNO both at 0.1 C, (320 mAhg-1 vs 284 mAhg-1) and at 1C (174 mAhg-1 vs 136 mAhg-1). This study demonstrated the viability of TNO/CMT for application as anode material for LIBs.

Topic: 17 - Advanced materials for sustainable energy (incl. nuclear fission and fusion, industrial gas turbines)
Abstract no. 2357

# Development of Silicon Carbide as a Nuclear Fuel Cladding


Presenting author: M CZERNIAK Luke
Affiliation: Westinghouse Electric Company
1332 Beulah Road,15235 Pittsburgh (Pennsylvania, United States)

Co-authors: LAHODA Edward (Westinghouse Electric Company), SIVACK Michael (Westinghouse Electric Company), LYONS John (Westinghouse Electric Company), BYERS William (Westinghouse Electric Company), WANG Guoqiang (Westinghouse Electric Company), XU Peng (Westinghouse Electric Company), OELRICH JR. Robert (Westinghouse Electric Company)



In response to the nuclear industry's desire for longer coping times following the Fukushima accident in Japan in 2011, General Atomics, as part of the Westinghouse EnCore?* accident tolerant fuel (ATF) program, is developing a silicon carbide (SiC) fuel cladding with the main goal of allowing the fuel within a nuclear reactor to tolerate the loss of active cooling for longer periods. Development of a SiC cladding that will improve the oxidation resistance of fuel cladding during a loss of active cooling is a focal point of study. SiC samples are being tested at the Westinghouse Churchill Facility and Massachusetts Institute of Technology (MIT) to provide out-of-pile corrosion and ultra-high temperature (>1600°C) testing and corrosion under irradiation analysis, respectively. The corrosion studies being carried out at Westinghouse occur in an autoclave where the SiC samples are exposed to a chemistry environment typical of a mid-cycle pressurized water reactor (PWR). Corrosion rates of composite samples are compared to alpha SiC and zirconium alloy controls. The ultra-high temperature tests analyze SiC samples for performance at temperature environments ranging from 1600-1900°C. In parallel, MIT is conducting irradiation tests of SiC samples in their research reactor. The corrosion under irradiation tests allow the SiC samples to receive a neutron dose to determine the mechanical properties and corrosion rates in a radiative environment. This study is being conducted to evaluate various SiC composites and manufacturing techniques to optimize a solution for an ATF cladding material.

Topic: 17 - Advanced materials for sustainable energy (incl. nuclear fission and fusion, industrial gas turbines)
Abstract no. 1979

# Focus on the thermal behavior of silicon carbide composites as accident tolerant fuel cladding

Presenting author: Dr DUQUESNE Loys
Affiliation: Framatome
10, rue Juliette Récamier,69003 LYON (France)

Co-authors:

Following the Fukushima-Daiichi accident in 2011, efforts have been made by nuclear industries to develop solutions to enhance accident tolerance of the nuclear reactors. The joint program of Framatome, CEA and EDF for the development of Accident Tolerant Fuel (ATF) led to consider SiC/SiC refractory composite materials as promising candidates to enhance the accident tolerance for current Light Water Reactor (LWR). One of the key challenges in the development of LWR cladding design consists in ensuring that the heat exchange between pellet and coolant is optimal. In this purpose, assessing the through-thickness thermal properties of SiC/SiC is essential for the cladding design.

The presentation will give a focus on the development of new methods to progress on the thermal characterization of SiC/SiC composites and the evolution of their thermal behavior regarding both the LWR environments and ATF requirements.

Topic: 17 - Advanced materials for sustainable energy (incl. nuclear fission and fusion, industrial gas turbines)
Abstract no. 2360

# Performance Assessment of the Westinghouse ENCORE Fuel with SiGA Cladding for PWRs


Presenting author: Dr XU Peng
Affiliation: Westinghouse Electric Company
1001 Pinnacle Point Drive,29223 Columbia (South Carolina, United States)

Co-authors: LAHODA Edwards (Westinghouse Electric Company), SIVACK Micheal (Westinghouse Electric Company), DECK Christian (General Atomics)



SiC/SiC composite cladding is a game-changing accident tolerant fuel (ATF) technology and a key component of Westinghouse's new EnCore? fuel. Funded by the Department of Energy in the United States, Westinghouse is partnering with General Atomics to develop the SiC cladding technology for LWRs. The lead test assembly (LTA) for EnCore? fuel with SiC/SiC composite cladding with lead test rods is planned for 2022. To meet this aggressive schedule, significant progress has been made to advance the SiC technology, and various developmental activities will be reported here. Four point bend testing and grid impact testing have been conducted to evaluate the seismic behavior of SiC fuel. Pressure drop testing, heat transfer testing, and critical heat flux testing will be conducted to determine the thermal hydraulic performance of the SiC fuel. Ultra-High Temperature tests (>1200°C) will be performed to assess the high temperature oxidation and degradation behavior of the SiC cladding. These performance based tests will be key to assessing the benefits of SiC cladding at normal operation conditions, design basis accidents, and in beyond design basis accidents.

Topic: 17 - Advanced materials for sustainable energy (incl. nuclear fission and fusion, industrial gas turbines)
Abstract no. 2249

# 2D Braided SiCf/SiCm Composite Tube for Nuclear Fuel Cladding


Presenting author: Dr CHEN Bo
Affiliation: Northwestern Polytechnical University
127 West Youyi Road,Beilin District,710072 Xi'an (Shaanxi, China)

Co-authors:



After decades of development in applications of aerospace thermostructures components, SiCf/SiCm composite is on the way to be mature for potential structural components such as the channel box of boiling water reactor (BWR), fuel cladding tube of light water reactors (LWRs), the control rod in gas cooled fast reactor (GFR), and other nuclear applications. Following the event at Fukushima in 2011, enhancing the accident tolerance of LWRs became a topic of serious discussion all over the world. Nuclear-grade SiC/SiC composite is the most promising alternative to the conventional commercial zirconium alloy in pressed water reactor (PWR) fuel cladding. The aim of the study is to examine the intrinsic microstructures and basic properties of the SiC/SiC composite fuel cladding tube for PWRs. Firstly, the two dimensional braided SiC fiber reinforced SiC matrix composite tube is prepared using the isothermal isobaric CVI process. Secondly, the X-ray computed tomography imaging analysis is used to characterize the tube internal structure nondestructively. And then, the tube is cut into several pieces and section analysis is also conducted using the scanning electric microscopy (SEM) assisted with the energy-dispersive X-ray spectroscopy (EDS) to further investigate the internal microstructures and determine the different substructures of the SiC/SiC composite. Finally, basic mechanical and thermophysical properties are evaluated.

Topic: 17 - Advanced materials for sustainable energy (incl. nuclear fission and fusion, industrial gas turbines)
Abstract no. 1763

# OVERVIEW OF SIC/SIC DEVELOPMENTS FOR CLADDING APPLICATION IN GAS FAST REACTOR


Presenting author: M SAUDER Cédric

Affiliation: DEN-Service de Recherches Métallurgiques Appliquées, CEA, Université Paris-Saclay, F91191 Gif-sur-Yvette, France
CEA SACLAY, bat 460 PC52,91191 Gif-Sur-Yvette (France)

Co-authors: BRAUN James (DEN-Service de Recherches Métallurgiques Appliquées CEA Université Paris-Saclay F91191 Gif-sur-Yvette France), LORRETTE Christophe (DEN-Service de Recherches Métallurgiques Appliquées CEA Université Paris-Saclay F91191 Gif-sur-Yvette France)



Ceramic Matrix Composites (CMCs) are largely studied as serious candidates for core applications in nuclear (fission or fusion) research programs. SiC/SiC composites are one of the most promising materials for these applications related to the high stability of SiC phase at high temperature under neutron irradiation. Nevertheless, the required final characteristics still represent a high challenge for scientific community.

Developments made in CEA for several years in the framework of Gas Fast Reactors program are described. A particular focus on fuel cladding application is proposed. First, the CEA "sandwich" concept and its advantages related to others will be presented in details. Work on the processing steps have conducted to the optimization of mechanical behavior. Many characterizations were achieved recently thanks to collaborative programs. For example, last results obtained in the frame of

European Materials Innovations for a Safe and Sustainable nuclear in Europe (MatISSE) demonstrate the excellent behavior of these materials under severe environments

Topic: 17 - Advanced materials for sustainable energy (incl. nuclear fission and fusion, industrial gas turbines)
Abstract no. 1942

# Evaluation of PVD Cr and Cr multilayer coatings on SiC for nuclear fuel cladding applications


Presenting author: Dr MOUCHE Peter
Affiliation: Oak Ridge National Laboratory
PO Box 2008, MS6138,37831-6138 Oak Ridge (TN, United States)
Co-authors: KOYANAGI Takaaki (ORNL), KATO Yutai (ORNL)



SiC/SiC ceramic matrix composites are a potential next generation LWR nuclear fuel cladding material due to their irradiation resistance, neutron transparency, and high temperature resistance. Corrosion behavior in boiling water nuclear reactor conditions, and fission gas release due to networked cracks are issues that still need to be addressed. 3-20?m thick Cr based coatings have been deposited on CVD and single-crystal SiC via high power impulse magnetron sputtering (HiPIMS) and b-field assisted or normal cathodic arc deposition by three different vendors. Cr, while introducing a manageable neutronics penalty, serves as a barrier to corrosion and a ductile layer to seal the rods. Titanium and CrN transition layers and multilayers were included in several of the HiPIMS samples as a method to alleviate detrimental mechanical coating/clad interactions. The deposition techniques were chosen due to the thickness requirements to minimize neutron absorption by the coating, as well as the dense morphology necessary to maintain hermeticity. This work compares the quality and robustness of the coatings from the different deposition methods, and focuses on the mechanical properties introduced by the additional layers. Coatings were characterized via XRD, GDOES, cross-sectional microscopy, nanoindentation, and scratch testing. Residual strain from the different deposition methods and transition layers was measured by sin2? XRD measurements. Cracks were initiated in the SiC substrates near the coating with a Vickers microhardness to examine crack propagation from the SiC to the coating.



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Topic: 17 - Advanced materials for sustainable energy (incl. nuclear fission and fusion, industrial gas turbines)
Abstract no. 2300

# Design Considerations for EnCore® ATF with SiC Cladding

Presenting author: Dr LONG Yun
Affiliation: Westinghouse Electric Company
1000 Westinghouse Dr.,16066 Cranberry Township (PA, United States)

Co-authors: CAI Lu (Westingouse Electric Company), XU Peng (Westinghouse Electric Company), LAHODA Edward (Westinghouse electric company), OELRICH Robert (Westinghouse electric company)

SiC is a promising ATF (accident tolerant fuel) cladding for improved high temperature performance during accident conditions over traditional Zr based material. However, SiC cladding can form micro-cracking from strong pellet and cladding mechanical interaction (PCMI) and lose hermeticity. This poses significant design challenges as fuel can expand and swell during normal operation and experience large instantaneous thermal expansions and fission gas swelling during transients. Several improvements to the traditional rod design and new design concept are introduced in this paper to mitigate the risk.
To maintain a traditional rod design, a large fuel-cladding gap has to be considered to mitigate PCMI. However, this will significantly increase fuel centerline temperature, especially in a low thermal conductivity fuel such as traditional UO2, resulting in a large fission gas release and/or swelling and also reduce the uranium loading resulting in a severe economic penalty. Alternative fuels like U3Si2 and UN are proposed for SiC cladding to reduce the centerline temperature, but a relative large gap is still needed. Liquid metals with high thermal conductivity can be used to fill the gap to improve gap heat transfer and lower fuel temperature. If selected correctly, this liquid material can fill in the cracks and either solidify or form a protective layer when in contact with coolant.
To maximize the benefit of SiC cladding, non-rod type fuel with SiC cladding is also evaluated to mitigate the PCMI risk between SiC cladding and fuel pellets. The goal is to reduce fuel temperature and reduce tensile stress during hard contact between the fuel and cladding and at the same time allow the use of UO2 fuel without compromising the economics of UO2.

Topic: 17 - Advanced materials for sustainable energy (incl. nuclear fission and fusion, industrial gas turbines)
Abstract no. 1835

# Thermal conductivity estimation of fully ceramic microencapsulated pellets with $ZrO_2$ as simulated particles


Presenting author: Mme DEWI ARIYANI KUSUMA
Affiliation: University of Fukui
3F, Honmachi 2 Chome 9-21,Tsuruga,9140051 Tsuruga (Fukui, Japan)

Co-authors: YAMAGUCHI Souichirou (University of Fukui), ONITSUKA Takashi (University of Fukui), UNO Masayoshi (University of Fukui)



Fully ceramic microencapsulated (FCM) fuel is manufactured by replacing the graphite matrix of conventional HTGR TRISO fuel compact with silicon carbide (SiC) matrix, because of SiC tremendous merits. SiC matrix on the FCM fuel not only increasing the barrier of fission product release, but also improving the heat transfer of nuclear fuel. Most of previous studies on FCM fuel thermal conductivity focused on the parameters of the SiC matrix characteristic and particle volume fraction. The present work deals with the estimation on the FCM concept fuel thermal conductivity with considering not only the particles size and volume fraction but also the matrix-particles interaction such as interfacial layer and gap formed between matrix and particle. $ZrO_2$(5wt.%$Y_2O_3$) particles were used to simulate the TRISO coated particle, and ?-SiC nanopowder as the matrix. The FCM pellets with various particle size and volume fraction were fabricated using spark plasma sintering (SPS) at 1800 degree C under uniaxial pressure of 8MPa for 15 minutes. Thermal conductivity of the pellets were investigated using laser flash analysis at the selected temperatures (100, 400, 700, 1000 and 1200 degree C). The experimental result showed that thermal conductivity of SiC-$ZrO_2$ pellets decreased with increasing particle volume fraction and temperature; the thickness of interfacial layer and gap increased with increasing particle size. Estimation of thermal conductivity has been conducted with Maxwell-Eucken model, Hasselman-Johnson model and modified of both model by considering the gas conductivity of the gaps between particle-matrix which might lower the effective thermal conductivity of pellets. The calculation result showed that modified model gave closest agreement to the measured thermal conductivity.

Topic: 17 - Advanced materials for sustainable energy (incl. nuclear fission and fusion, industrial gas turbines)
Abstract no. 1830

# Direct Decomposition of Nitrous Oxide to Nitrogen and Oxygen Using C-type Ytterbium Oxide-Cobalt Oxide Catalysts


Presenting author: Dr NUNOTANI Naoyoshi
Affiliation: Department of Applied Chemistry, Faculty of Engineering, Osaka University
2-1 Yamadaoka,565-0871 Suita (Osaka, Japan)

Co-authors: CHO Chang-Min (Department of Applied Chemistry Faculty of Engineering Osaka University), IMANAKA Nobuhito (Department of Applied Chemistry Faculty of Engineering Osaka University)



Nitrous oxide formed by chemical processes has received attention, because it causes global warming and ozone layer depletion. To decrease nitrous oxide emission, we developed advanced catalysts of ytterbium oxide-cobalt oxide with C-type rare earth sesquioxide structure for the direct nitrous oxide decomposition, where C-type structure possesses large interstitial open spaces for the nitrous oxide adsorption. The introduction of Co2+/3+ ions into the ytterbium oxide lattice resulted in an improved catalytic activity due to the enhancement in redox properties and the increase in amount of active sites. Among the samples prepared, (Yb0.9Co0.1)2O3 shows the highest catalytic activity, and nitrous oxide was completely decomposed to nitrogen and oxygen gases at 500 degree C.

Topic: 17 - Advanced materials for sustainable energy (incl. nuclear fission and fusion, industrial gas turbines)
Abstract no. 2631

# A new silicon carbide matrix for fully ceramic microencapsulated fuels


Presenting author: M ROHIT Malik
Affiliation: Functional Ceramics Laboratory, Department of Materials Science and Engineering, The University of Seoul
,02504 Seoul (South Korea)

Co-authors: KIM Hyun-Min (Functional Ceramics Laboratory Department of Materials Science and Engineering The University of Seoul Seoul 2504 Republic of Korea), KIM Young-Wook (Functional Ceramics Laboratory Department of Materials Science and Engineering The University of Seoul Seoul 2504 Republic of Korea), LIM Kwang-Young (Materials Development Section KEPCO Nuclear Fuel Daejeon 34057 Republic of Korea)



Fully ceramic microencapsulated (FCM) fuels consist of microencapsulated tristructural isotropic (TRISO) particles embedded in an impervious SiC matrix. The SiC matrix in the FCM fuels offers various advantages including good thermal conductivity, radiation damage resistance, environmental stability, and proliferation resistance. Furthermore, enveloping TRISO particles within a dense SiC matrix provides multiple barriers to fission product release. The objective of this study is to report a new quaternary additive composition (AlN-Y2O3-Sc2O3-MgO) for the successful densification of a SiC matrix at 1850 °C without applied pressure. Typical flexural strength, fracture toughness, and hardness of the SiC ceramics sintered at 1850 °C without applied pressure were determined as 510 MPa, 6.9 MPa?m1/2, and ~25 GPa, respectively.

Fully ceramic microencapsulated (FCM) fuels containing ~37 vol% TRISO particles could be successfully sintered at 1850 °C using the above matrix without applied pressure. TRISO particles were not damaged during processing, which included cold isostatic pressing under ~200 MPa and sintering at 1850 °C for 2 h in an argon atmosphere. The thermal conductivity of the pressureless sintered FCM pellet with 37 vol% TRISO particles was ~44 Wm-1K-1 at room temperature.

Topic: 17 - Advanced materials for sustainable energy (incl. nuclear fission and fusion, industrial gas turbines)
Abstract no. 2245

# Fracture behavior of uncoated SiC woven fabric composites with SiC matrix incorporating BN particles


Presenting author: Dr SHIMODA Kazuya
Affiliation: National Institute for Materials Science
1-2-1,Sengen,3050047 Tsukuba (Ibaraki, Japan)

Co-authors: HINOKI Tatsuya (Kyoto University), KAKISAWA Hideki (National Institute for Materials Science)



SiC woven fabric composites lacking a fiber/matrix interface and incorporating BN particles in SiC matrix were newly developed using sandwiched layers with SiC woven fabrics and SiC-based matrix sheets in a laminate to achieve crack deflection. Since final fracture of composites is generally caused by cumulation of the microfractures, initial fracture is recognized as the important key to understand the mechanical properties. In this study, initial fracture in plain and satin woven fabric composites were considered. Initial fracture behavior in textile composites were decided by the effect of weaving structure. Geometry of textile fabric, that is, crimp ratio and aspect ratio were investigated and effects of the geometry on the proportional point on stress-stain curve and fracture behavior were measured. Initial fracture in composites both transverse crack and filament fracture was simultaneously observed. The effects of changes in crimp ratio and aspect ratio on the fracture of woven fabric were provided and discussed.

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# OVERALL CONFERENCE SCHEDULE

| Time | Sunday, Sept. 22 | Monday, Sept. 23 | Tuesday, Sept. 24 | Wednesday, Sept. 25 | Thursday, Sept. 26 |
|---|---|---|---|---|---|
| 8:00 | | Registration | Registration | Registration | Registration |
| 9:00 | | Opening; Plenary | Plenary | Plenary | Plenary |
| 10:00 | | Coffee break | 5, 10, 7, 11, 14 | 8, 9, 1, 2, 16 | 8, 17, 1, 4, 15 |
| 11:00 | | 5, 13, 12, 11, 3 | Coffee break; 5, 9, 7, 11, 14 | Coffee break; 8, 9, 1, 4, 16 | Coffee break; 8, 17, 1, 4, 15 |
| 12:00–13:00 | | Lunch | Lunch | Lunch; Poster session | Lunch |
| 14:00–15:00 | | 5, 13, 12, 11, 3 | 5, 9, 7, 2, 14 | Poster session | 8, 17, 1, 4, 6 |
| 16:00 | Registration | Coffee break | Coffee break | Bus to Bordeaux | Coffee break |
| 17:00 | Registration | 5, 10, 12, 11, 7 | 8, 9, 7, 2, 16 | Bordeaux tour | 8, 1, 4, 6 |
| 18:00 | Welcome reception | 5, 10, 12, 11, 7 | 8, 9, 7, 2, 16 | Bordeaux tour | Concluding remarks |
| 19:00 | Welcome reception | | | Gala dinner | |