HT-CMC/10th

Bordeaux 22-26 SEPT. 2019

10th International Conference on High Temperature Ceramic Matrix Composites

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BOOK OF ABSTRACTS

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

September 22-26, 2019

BORDEAUX CONGRESS CENTER, FRANCE

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Oral presentations

(Order by day and by room)

Some abstracts are missing. They will be added after the conference.

Monday 2019-09-23

Amphi A

Plenary

Monday 2019-09-23

Amphi A

Topic 5 – Non-oxide CMC S

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.

EBERLING-FUX Nicolas
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.

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

XU Zeshui
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 SiC_f/SiC with low fiber volume fraction were 2.45 g/cm^3 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 SiC_f/SiC.

Key words: SiC chopped fibers; Quasi-isotropic SiC-based composites; Mechanical properties

Evaluation of property and performance of SiC_f/SiC composites

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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 SiC_f/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-SA3^{TM} 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-SiC_f/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 SA3^{TM}) 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 β-SiC powder (4620 KE, Nano Amor Inc., USA) with 12 wt.% Al_2O_3-Y_2O_3 sintering additive by AC- EPD. Twenty layers of SiC fabric with β-SiC powder embedded in the matrix using EPD were stacked to carry out the hot pressing at 1750^{o}C and 20 MPa for 2 h under Ar atmosphere. The oxidation behaviors of EPD SiC_f/SiC were evaluated by ablation test. The ablation temperature range is 1300^{o}C ~ 2000^{o}C and the oxidation time is 30 min. To investigate the oxidation behaviors, weight change measurement, microstructure observation, and phase analysis were conducted.

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

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The elaboration of $TiSi_2$/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 $TiSi_2$ 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 $Ti_{0.16}Si_{0.84}$ alloy in SiC+TiC compacts at 1380°C. The compacts were prepared from mixtures of micronic SiC (α or β 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-$TiSi_2$ composites is complex but could be possible in limiting or in prohibiting the activity gradients. Finally, some improvements are proposed.

Keywords: SiC; Alloy; Reactivity; Infiltration; Thermodynamic; Composites

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

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Abstract
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-venyl-benzene (DVB). The impregnated preforms were consolidated/processed by heating them up to 300ºC under pressure to obtain fiber volume fraction (V_f) of the order of 55%. The consolidated composites were pyrolyzed up to 1600ºC and 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/cm^3. 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 and 350-450 MPa respectively. While, for the 2D C/SiC composites, flexural and tensile strength were found to be 300-350 MPa and 150-230 MPa respectively. The properties were greatly influenced with the process parameters such as, V_f, interface characteristics and pyrolysis temperature. The strength was highest for composites heat treated at 1400ºC and exhibited increasing trend with increasing V_f and 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 2.5×10^{-7} to 2.2×10^{-6} m/m ºC in temperature range of 200 to 1000ºC. Thermal diffusivity decreases with temperature from 32 mm^2/s to 7 mm^2/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.

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

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A hybrid Microwave assisted Chemical Vapor Infiltration (MW-CVI) pilot plant to produce silicon carbide-based ceramic matrix composites (SiC_f/SiC) was designed, built and setup, as a part of the European project HELM[1]. 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.

Keywords: SiC/SiC, innovative design, advanced processing technique, manufacturing technology

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[1] http://www.helm-project.eu/

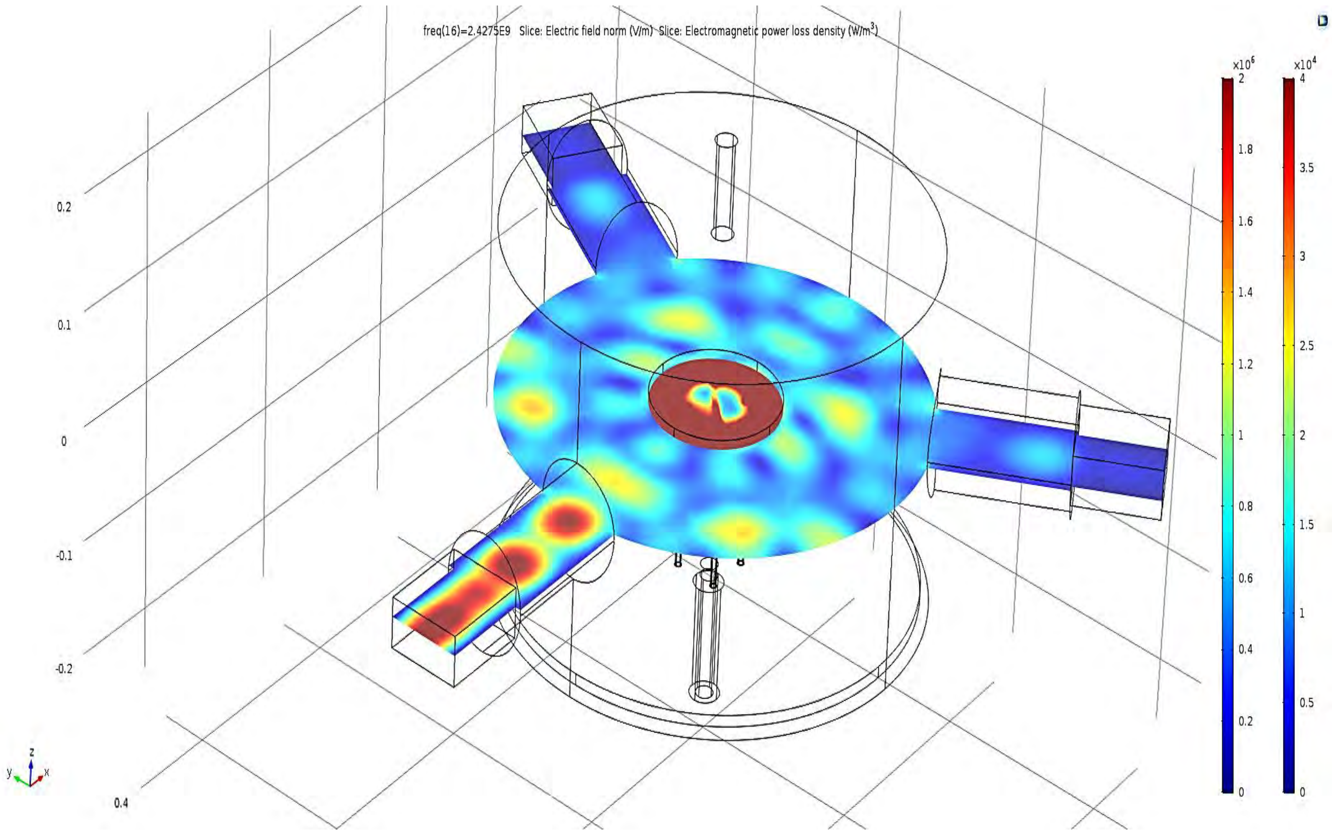

Figure 1 – *Electric field distribution [V/m] in the plane of the sample and Electromagnetic power released in the sample [W/m^3]*

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

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Chemical vapor deposition SiC 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).

Keywords: monomethylsilane, SiC CVI, direct resistance heating

Monday 2019-09-23

Parallel room H1+H2

Topic 13 – Joining

Wetting and phase interaction between C_f/SiC and transition metal disilicides-based alloys

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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 C_f/SiC composites. The wetting and phase interaction of molten transition metal disilicides ($ZrSi_2$ and $HfSi_2$) 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 C_f/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 C_f/SiC substrates and various different transition metal disilicides-based alloys (containing B_4C, 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 ZrB_2(HfB_2)-SiC-RE coating layer on the surface of C_f/SiC.

Monday 2019-09-23

Parallel room H1+H2

Topic 10 – Carbon-Carbon Composites

Ablation response of ZrC-SiC inhibited carbon/carbon composites subjected to oxyacetylene torch

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Introduction

Extensive effort has focused on the development of the unique ablation resistance of C/C composites. It is a favored route to improve the ablation resistance of C/C composites by adding the UHTCs into the carbon matrix of the composites. Reactive melt and infiltration (RMI) process as an efficient technique is desirable to suffice the combination of high performance and low cost. Plate shaped sample of the C/C composites inhibited by ZrC-SiC (C/C-ZrC-SiC) have shown a desired ablation resistance performance after the oxyacetylene torch (OAT) testing. However, the ablation response of the wedge shape sample of C/C-ZrC-SiC composites by RMI should be investigated due to unique characteristics of the reaction-bonded matrix. The inhibited composites is designed as the wedge shape which is capable of exhibiting their response to the ultrahigh temperature and mechanical scouring of the flame stream.

In the present study, the C/C-ZrC-SiC composites were prepared by RMI. To reflect the ability of the two systems to resist the ultrahigh temperature and mechanical scouring of the flame stream, the samples were designed for the wedge shape. The C/C composites were used as the reference for comparison.

Materials and Methods

The porous C/C preform was selected for the preparation of C/C-ZrC-SiC. The preforms were in advance machined into the wedge shape and followed by RMI of ZrSi powder at 2100 °C under Ar atmosphere, held for 2h, resulting in formation of C/C-ZrC-SiC composites (Fig. 1a). Ablation testing was performed using an oxyacetylene torch with the heat flux of 2.38 MW/m^2 for 120 s. The compositions and microstructure of the sample were characterized by X-ray diffraction and scanning electron microscopy.

Results and Discussion

The reaction-bonded ZrC-SiC matrix is developed in the fiber webs and exhibits a dense structure after RMI process, as shown in Fig. 1 (b) and (c), respectively. Exposing the samples to the torch, the tip of the sample undergoes the ultrahigh temperature associated with the intense mechanical scouring of the flame stream. Therefore, thermal load and mechanical scouring are the two quite severe challenges on the wedge-shaped sample. The tip temperature of C/C-ZrC-SiC composites is as high as 2485 ºC (Fig. 2a), which is significantly higher than that of the plate shaped sample at the identical heat flux due to the shape effects. For C/C sample, the tip temperature was maintained at 1735 ºC (Fig. 2a). After testing, the tip and surface of

C/C-ZrC-SiC sample is oxidized by ultrahigh temperature. However, the sample retains a complete shape and no spallation occurs, indicating that the ZrO_2 at the tip of sample possesses a high configurational stability and is favorable to play a thermal barrier, endure and resist the high pressure and mechanical denudation of the flame stream. The ablation resistance performance of C/C-ZrC-SiC is significantly superior to that of C/C. The mass and linear ablation rate of C/C is caused by oxidation of carbon matrix and carbon fibers. For C/C-ZrC-SiC sample, the mechanical denudation of the flame stream acting on the sample surface is weakened by the blocking of the tip and the surface is mainly affected by the ultrahigh temperature oxidation. Microstructure of the surface from the region near the tip to the tail is evolved due to presence of the temperature gradient. The porous ZrO_2 is present in the region near the tip due to the full depletion of SiC, carbon fiber and matrix by oxidation, as shown in Fig. 2b. The middle region consists of the outer ZrO_2 layer and ZrO_2-SiO_2 sub layer. The grain size of ZrO_2 in the outer layer is large compared to the sub layer due to the depletion of SiO_2 in the outer layer, as shown in Fig. 2c. At the tail, the depletion of SiO_2 is decreased and the SiO_2 is spread on this region, as shown in Fig. 2d.

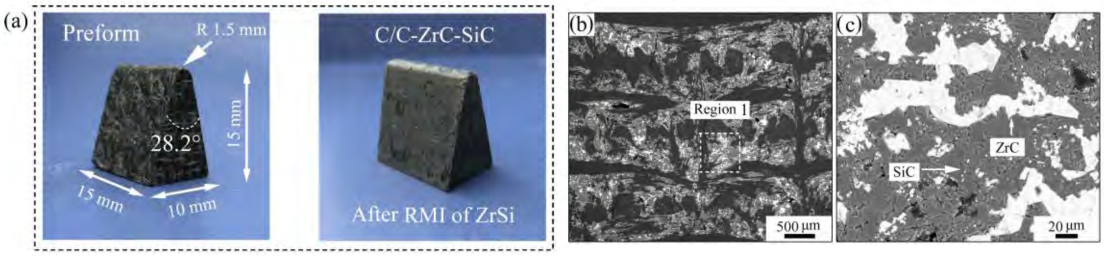

Fig. 1. (a) Shape and size of the sample, (b) cross-sectional backscatter-SEM overview of C/C-ZrC-SiC and (c) magnification of the region 1

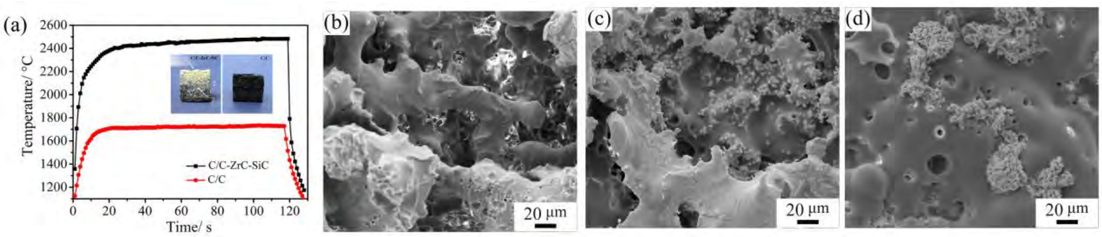

Fig. 2. (a) the tip temperatures of C/C-ZrC-SiC and C/C samples, (b) the region near the tip, (c) the middle region and (d) the tail of C/C-ZrC-SiC

Conclusions

Wedge-shaped samples of C/C-ZrC-SiC composites were fabricated by RMI of ZrSi powder and tested using an oxyacetylene torch 2.38 MW/m^2 for 120s. The tip temperature of C/C-ZrC-SiC was up to 2485 ºC. C/C-ZrC-SiC sample experienced high temperature gradient and showed a shape integrity after ablation. Microstructure evolution was occurred from the region near the tip to the tail due to the effects of temperature gradient.

Acknowledgment

This work has been financially supported by the National Natural Science Foundation of China under Grant Nos. 51472202 and 51521061.

The ablation behaviors and mechanism of C/C-SiC-ZrC composite by CO2 laser

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C/C-SiC-ZrC composite was prepared by chemical vapor infiltration (CVI) combined with Si0.87-Zr0.13 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.

Keywords: laser ablation; UHTCs; C/C-SiC-ZrC; finite element analysis

Monday 2019-09-23

Parallel room F1+F2

Topic 12 – NDT and Health Monitorings

In situ X-ray microtomography characterisation of mechanical damage and failure in plain weave SiC/SiC composites

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Abstract. 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.

Keywords: Composites, ceramic matrix composites, damage mechanism, X-ray computed tomography, non-destructive testing

Research progress on non-destructive testing of ceramic matrix composites

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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.

Keyword: NDT, Defects, CMCs

Preferred session : NDT and health monitoring

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Understanding Damage Mechanisms of CMC Blade Root Sub-elements

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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.

Measurement of the electrical resistivity to monitor the oxidation propagation inside damaged SiC/SiC composites during ageing under oxidizing environments

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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.

Real-time non-destructive damage evaluation of C/SiC composites during fatigue loading

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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. This paper investigated infrared thermography (IR thermography) and acoustic emission (AE) with the aim of providing an in situ characterization technique of damage under fatigue loading of the 2D plain woven C/SiC composites specimens.

Plain plate and center-holed specimens were performed on Instron8801 servohydraulic fatigue machine under load control at stress ratio ($\sigma_{min}/\sigma_{max}$) of 0.1 and sinusoidal frequency of 10Hz. IR thermography was recorded using an infrared camera (FLIR ThermaCAM SC3000) with 320×240 pixels, which provided high sensitivity of less than 0.02K at 300 K at high speed frame rate. AE signal was detected by two acoustic emission wide band sensors attached on specimen, and an AE 2ch. DAQ system (PAC PCI-2 system) was used to record and store the AE wave forms. The infrared camera and AE 2ch. DAQ system were triggered with the +12V voltage to synchronously record the data when the test started.

Damage evolution was discussed on the basis of the calculation results of the modulus. Modulus degraded of the composite during fatigue loading, which was attributed to micro-cracking and fiber fracture. Thermal dissipation Q was deduced based on the first law of thermodynamics, which 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 a steady value as the proceeding cycles. When the applied stress exceeded the endurance fatigue limit, Q rose quickly until led to failure of the composites. AE hits and energy was discussed based on calculation results of the AE data. Higher applied stress would cause more damage of composites, and more AE signals were detected. Compared with modulus, Q and AE energy had fairly well agreement with the damage evolution. It can be concluded that it is possible to employ these non-destructive evaluation methods as in-situ damage evolution indicators for 2D C/SiC composites.

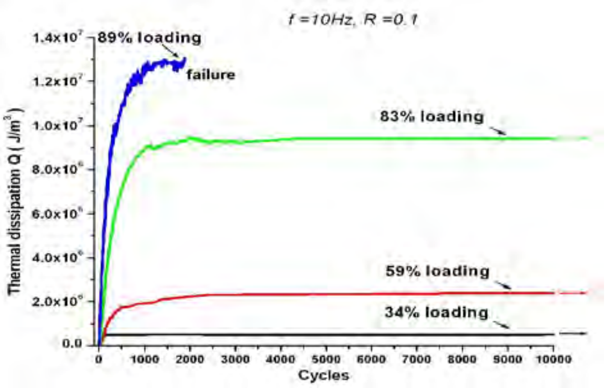

Fig.1 Thermal dissipation varied with stress levels

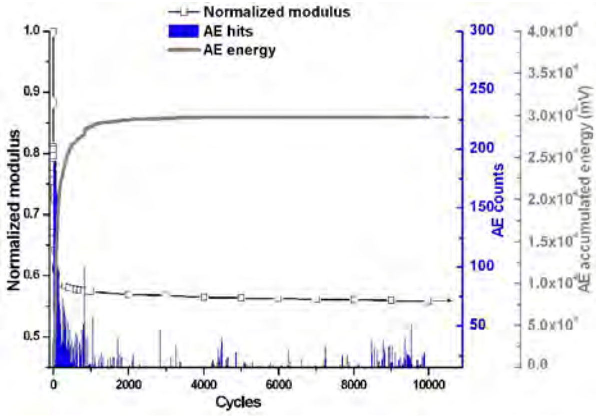

Fig.2 AE signals varied under83%loading level

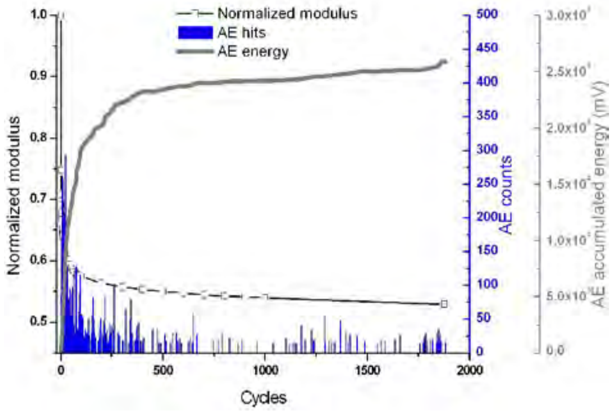

Fig.3 AE signals varied under89%loading level

Keywords: real-time, infrared thermography, acoustic emission, damage evaluation, 2D plain woven C/SiC composites

Key reference

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Monday 2019-09-23

Parallel room E1+E2

Topic 11 – Thermomechanical behavior and performance

Microstructure and damage evolution of SiC_f/PyC/SiC and SiC_f/BN/SiC mini-composites: A synchrotron X-ray computed microtomography study

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ABSTRACT

Continuous SiC fibre-reinforced SiC matrix composites (CMCs) are promising thermal-structure materials for advanced aeronautics and space applications due to their low densities, high toughness, high strengths, good oxidation resistances, and high durability at elevated temperatures. SiC_f/PyC/SiC and SiC_f/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 SiC_f/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 SiC_f/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 SiC_f/PyC/SiC composites as the oxidation temperature increases to 1600 °C. On the other hand, the oxidation products of B_2O_3 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 B_2O_3, the SiC_f/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 SiO_2 grain growth from room to high

temperatures. This work provides guidance for predicting the service life of SiC_f/PyC/SiC and SiC_f/BN/SiC composite materials, and is fundamental for establishing multiscale damage models on a local scale.

Keywords:

SiC_f/PyC/SiC; SiC_f/BN/SiC; mini-composites; synchrotron X-ray computed microtomography; microstructure; damage evolution

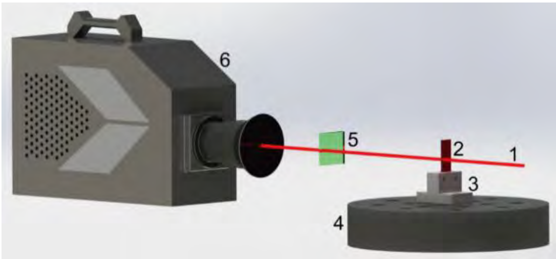

Fig. 1. Schematic diagram of the CT setup. 1: X-ray beam; 2: sample; 3: standard kinematic mount; 4: high-precision rotation stage; 5: scintillator; and 6: camera.

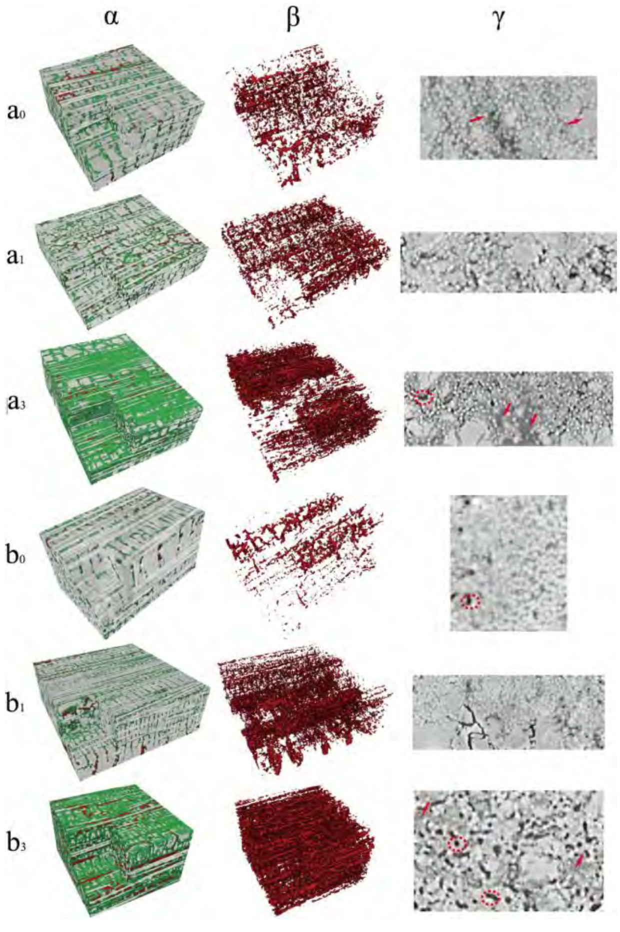

Fig. 2. Reconstructed 3D images (left column) and corresponding 2D projections (right column) of the SiC_f/PyC/SiC and SiC_f/BN/SiC composites under an air oxidation atmosphere of different temperatures. The middle column is the 3D distribution of the void in the sample. The grey, red, and green colours represent the SiC, void, and SiO_2 respectively.

STUDY OF DAMAGE OF COMPLEX WEAVED CMC STRUCTURES SUBMITTED TO MULTI-AXIAL THERMO-MECHANICAL LOADING BY AN IN-SITU EXPERIMENTAL APPROACH

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Key words : micro-tomography, Ceramic Matrix Composite (CMC), local damage, local mechanical properties, complex weaved structure

Abstract

Ceramic matrix composites maintain good mechanical properties at very high temperature, and so are promising materials for hottest parts of aircraft engines. Due to their complex structure, it is important to observe, understand and model local thermo-mechanical behaviour of parts, especially in singular zones. That is why an original full-field measurement approach, including the conception of an in-situ device, has been developed to study complex samples submitted to representative service loadings.

1 Introduction

3D-weaved SiC/SiC Ceramic Matrix Composites (CMCs) are expected to replace super-alloys in aircraft engines. The weaved-structure is designed to match part geometry and loading, including complex zones. A good understanding of the thermo-mechanical behaviour of those zones, based on experiments, is needed to model, to scale and to certify aeronautical parts. The multi-scale, inhomogeneous and anisotropic nature of the material requires innovative and heavily instrumented tests that can feed models based on the real structure of the material [1, 2]. During in-situ tests, X-ray tomography, coupled with Digital Volume Correlation (DVC) [3, 4], gives access to displacement field in the bulk of the sample. Tensile tests have already demonstrated the capability of such an approach [5], namely on CMCs at high temperature [6, 7]. A new test device have been designed to study, at temperature up to $1,000°\mathrm{C}$, loadings that represent service conditions of parts. Full-field measurement provides data to identify, with a single test, local thermo-mechanical properties that would have been inaccessible through a conventional approach. The objective is to build a model based on physical observations and to study damage as it is the critical criterion to design such parts.

2 Experimental Method

Samples are designed to optimise multi-axial loading using a single mechanical actuator (Fig. 1). T-Shape and corner samples are proposed with complex weaving structures and dimensions optimised for the tomography set-up. The multi-axiality of the loading in the area of interest is induced by the geometry of the samples and the thermal gradient applied between the sample sides. Tests are quasi-static and performed at thermal equilibrium because the measurement of the thermal field during the tomographic acquisition is difficult to instrument. The loading is applied by steps between each tomography acquisition. To define acquisition steps, an acoustic emission sensor which detects the occurrence of cracks, is used. The displacement field between two steps is measured by DVC. The thermal field is obtained by combining a thermal camera, a pyrometer and a thermocouple.
The loading device (Fig. 1) intersects the X-ray beam during the acquisition. To correct the artefacts due to columns' shadow, a reconstruction algorithm taking into account missing angles has been developed [8]. It allows the use of the projected integrated-DVC algorithm.

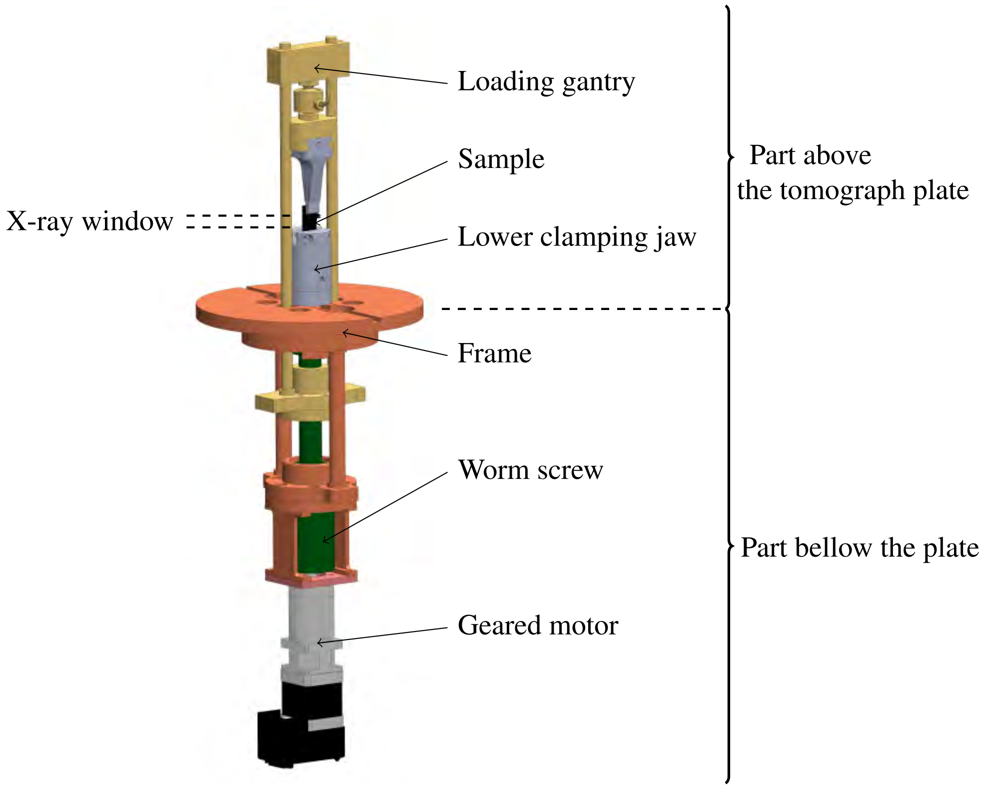

FIGURE 1 – Scheme of the experimental device. Load is applied by a gantry which passes through the tomograph rotating plate so that the actuator is deported under the plate to reduce imbalance and free the observation zone.

3 Conclusion

The study of complex and hardly accessible zones of CMC parts requires the developement of a new specific device which is versatile enough to perform several tests in various conditions. It will be the support of the implementation of innovating approachs such as projection-based DVC [9]. Tests will be carried out soon on SOLEIL PSICHE beam-line.

Acknowledgements

This work is supported by a convention CIFRE implying *Association Nationale de la Recherche et de la Technologie* and Safran Ceramics. Safran group and *Direction Générale de l'Aviation Civile* are acknowledged for funding it through PRC MECACOMP.

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In-situ observation of contact damage in a SiC/SiC Ceramic Composite

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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.

Key Words: SiC-SiC Composite, Contact Damage, Indentation, X-ray Computed Tomography, Digital Volume Correlation.

Monday 2019-09-23

Parallel room D1+D2

Topic 3 – Interphases

OPTIMIZATION OF THE SIC/SIC COMPOSITES FIBER/MATRIX INTERFACES FOR NUCLEAR APPLICATIONS

(Proposal abstract for topic 3 "Interphases")

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Abstract 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.

Keywords SiC/SiC composites, interphase, nuclear, pyrocarbon, mechanical behavior.

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Rare-earth disilicate fiber coatings for SiC/SiC CMCs

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Rare-earth disilicates ($RE_2Si_2O_7$) 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 $Y_2Si_2O_7$ interface were exposed to steam and tensile tested. The effect of steam on the functionality of the $Y_2Si_2O_7$ interface coatings was studied and will be reported.

ENHANCEMENT OF INTERFACE INTERACTIONS OF SiC FIBER REINFORCED COMPOSITES IN COOPERATION WITH ‘FUZZY FIBER’ BNNTs COATING ONTO SiC FIBER.

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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 (SiC_f)-SiC matrix will play a significant role on the final properties of overall structure. Hence, coating SiC_f with an interphase layer has been adopted in literature to enhance the fiber/matrix (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 SiC_f 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 SiC_f 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 SiC_f. 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.

Monday 2019-09-23

Parallel room D1+D2

Topic 7 – UHTCS & MAXS

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

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Keywords: CVD, SPS, RMI, Carbides, protection against oxidation.

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 and most of metallic carbides 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) presenting a better oxidation resistance at lower temperatures.

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 of coatings and the third one is the Reactive Melting Infiltration of $HfSi_2$.

On first hand, this study describes the CVD conditions for the deposition of HfC from the metallic hafnium pellets to get hafnium chlorides followed by the reduction of the chlorides by H_2 and the deposition of HfC with the methane as carbon precursor. This 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]. This alternated microstructure can even be infiltrated inside a 3D C/C.

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. This powder is then sintered on top of a C/C composite. The sintering conditions to obtain an uncraked coating will be presented as well as microstructures [2].

In a third way, R M I enables, at the fusion temperature of $HfSi_2$ (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. Between each RMI a pitch infiltration followed by a pyrolysis is necessary to get some C inside the C/C sample [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. As they are thicker the coatings obtained by SPS had been used to understand the oxidation mechanism involved during oxidation [4]. In terms of protection the CVD coatings enable to protect a C/C during 200 seconds at 2000°C and 370 seconds at 1900°C.

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

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2. Xi'an Aerospace Composites Research Institute, Xi'an 710025)

Abstract: 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/cm^3) 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/m^2) 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.

Key words: Carbon/carbon, carbon/ceramic, oxy-acetylene test, chemical vapor infiltration, precursor infiltration and pyrolysis

Tuesday 2019-09-24

Amphi B

Plenary

Multiple-criteria decision analysis for reliability assessment of CMCs

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The problem of reliability of CMC is still unsolved and developing accurate reliability assessment methods for CMC becomes an important research topic. Various conventionally available evaluation methods, such as thermography, X-ray CT etc… have been utilized to monitor the degradation level of various kinds of CMCs. However, the degradation behavior of CMCs is affected by irregular micro-scale damage evolution phenomena controlled by both physical and chemical factors that are hard to be predicted. In the present talk, some new methods: Talbot-low X-ray CT, GHz microwave spectroscopy, vibration resonance image analysis, etc… are addressed. To achieve a better reliability assessment of CMCs using a set of results from these new techniques and from traditional approaches, an AI-based multiple criteria decision analysis is introduced. Some examples will be presented. Problems related to life prediction from accelerated degradation tests are also explained from a reliability assessment point of view.

Tuesday 2019-09-24

Amphi B

Topic 5 – Non-oxide CMC S

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

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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 techniques [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 CRIM[2] jointly with specialists of the CIAM[1] have developed various composite design 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.

Figure 1 - Nozzle apparatus from DCS "Skeleton"

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

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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 its high strength and creep resistance at high temperatures, high thermal conductivity, and high melting point, tungsten (W) would be the ideal material for this application. However, tungsten is brittle at room temperature and prone to operational embrittlement by overheating and/or neutron irradiation. Tungsten fibre-reinforced tungsten composites (W_f/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/m^2. Highlights are the development of a continuous manufacturing routine for CVD and mechanical characterisation with in situ synchrotron tomography.

As the matrix material in W_f/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 W_f/W and W fibres.

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

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Abstract:

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.

Keywords:

Rapid densification; Carbon fibre-reinforced silicon carbide; Infiltration-assisting holes; LA-CVI

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

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Abstract:

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.

Keywords:

SiC/SiCN, CT, combustor test, combustion, effusive cooling, SiC coating

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

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Abstract

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.

Keywords

C/C-SiC - Machining - Surface – Strength – CMC – Ceramic Matrix Composite – Roughness - Milling

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

Laifei Cheng, Fang Ye, Zhiwei Gao, Naiqi Chen

Abstract

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·m$^{1/2}$. 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·m$^{1/2}$ 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.

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

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（Xi'an Aerospace Composite Materials Research Institute）

Abstract：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, microstructure 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 chemical vapor infiltration and resin impregnation/carbonization (CVI+PIC) methods with the density of 1.35~1.45g/cm^3. Then, the C/C-SiC composites were prepared by RMI. The capillary infiltration test of C/C porous preforms was carried out using water instead of liquid silicon. And the microstructure and bending behavior at high temperature(1200~1800℃) of the C/C-SiC composites were discussed. 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 μ-CT is 0~0.13mm, in which the pore size of the small pore(less than 30μm) 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. The residual silicon is solid at 1200~1400℃, which has good bearing capacity and stress transfer ability. So the C/C-SiC composites have high bending strength and low toughness. The residual silicon is converted into liquid state at 1600~1800℃, and the porosity of the composites increases. So the strength of the C/C-SiC composites decreases, the toughness increases.

Tuesday 2019-09-24

Amphi B

Topic 8 – EBC/TBC

Enhanced EBC Development and Behaviour Analysis for High Temperature CMC Components

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Keywords : Silicon Carbide, Ceramic Matrix Composites, Environmental Barrier Coating.

Abstract :

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.

Tuesday 2019-09-24

Parallel room H1+H2

Topic 10 – Carbon-Carbon Composites

Tuesday 2019-09-24

Parallel room H1+H2

Topic 9 – PDCS

Phase and nanostructure evolution in Zirconium modified polymer derived silicon carbonitride ceramic hybrids

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Abstract:

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-ZrO_2 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 Si_3N_4 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 ZrO_2 in the ceramic matrix may help achieve enhanced toughness of the bond coat.

(Keywords: *Polymer Derived Ceramics; SiCN; ZrO_2; EBC; HRTEM)*

Tuesday 2019-09-24

Parallel room F1+F2

Topic 7 – UHTCS & MAXS

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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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.

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

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Institute of Solid State Chemistry and Mechanochemistry SB RAS, Novosibirsk, Russia

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 $Ta(Hf)Ir_3$. 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.

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

Vinothini Venkatachalam[1*], Jon Binner[1]
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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 (TPS).

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 (unsintered) zirconium diboride (ZrB_2) powder and with a matrix of carbon. 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.

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

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Abstract

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 C_f reinforced ZrC matrix composites. A suitable interphase of SiC combined with rare earth oxide (CeO_2 & Y_2O_3) 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.

Key words: UHTCMCs, zirconium carbide, silicon carbide, rare earth oxides, radio frequency chemical vapour infiltration

Preferences:
1: TOPIC 7- UHTCs and MAX phases
2: TOPIC 2- Fibers and Preforms
3: TOPIC 14- CMC Applications in Space Transportation

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

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Abstract. Over the past two decades, a class of ternary transition metal carbides or nitrides known as M_nAX_{n+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 Fe_2AlB_2 [3] and MoAlB [4] (both MAB phases) showing high fracture toughness, damage tolerance and oxidation resistance.

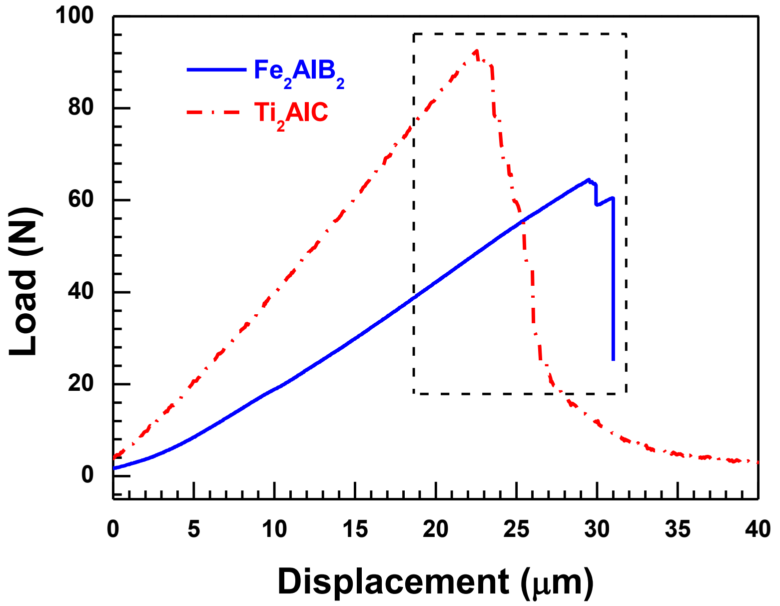

Figure 1 Plot of load vs displacement during SENB of Fe_2AlB_2 and Ti_2AlC [3]

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 Fe_2AlB^2 bulk for the first greatly reduced the fabrication time of MAB phases [3]. No dominant indentation cracks are observed, indicating that Fe_2AlB_2 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].

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Carbosilicothermic synthesis of MAX phases and high-entropy MX carbides

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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 Ti_4SiC_3 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, $(Ti_{0.2}Zr_{0.2}Hf_{0.2}Nb_{0.2}Ta_{0.2})C$ has been obtained by this method.

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

Oxidation in air plasma of ZrB_2 and HfB_2-SiC based composites from 1800 to 2200 K

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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.

Keywords: UHTC; testing; high temperature; air plasma; oxidation; emissivity

Retained strength of UHTCMCs treated above 2273 K in oxidizing environment

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A new class of non-oxide ultra-high temperature ceramic-matrix composites (UHTCMCs), currently under study within the Horizon 2020 European C^3HARME 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.

Keywords: UHTCMC, thermal protection systems and hot structures, propulsion systems for spacecraft, self-healing.

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

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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 [1, 2], 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 [3, 4]. Since monolithic materials demonstrated low fracture toughness, low thermal shock resistance and lack of damage tolerance [5], fiber-reinforced Ultra-High-Temperature Ceramic Matrix Composites (UHT-CMC) [6, 7] are currently regarded as the most promising technology for overcoming state-of-art materials limitations [8, 9]. Hence, the Horizon 2020 European C^3HARME research project [10] 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 [11].

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 (Fig. 1); 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 (Fig. 2). 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.

Keywords: UHT-CMC; Arc-jet testing; Hybrid rocket nozzles

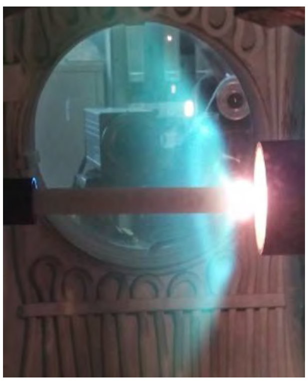

Fig. 1 Picture taken during test on UHT-CMC specimen in arc-jet supersonic wind tunnel.

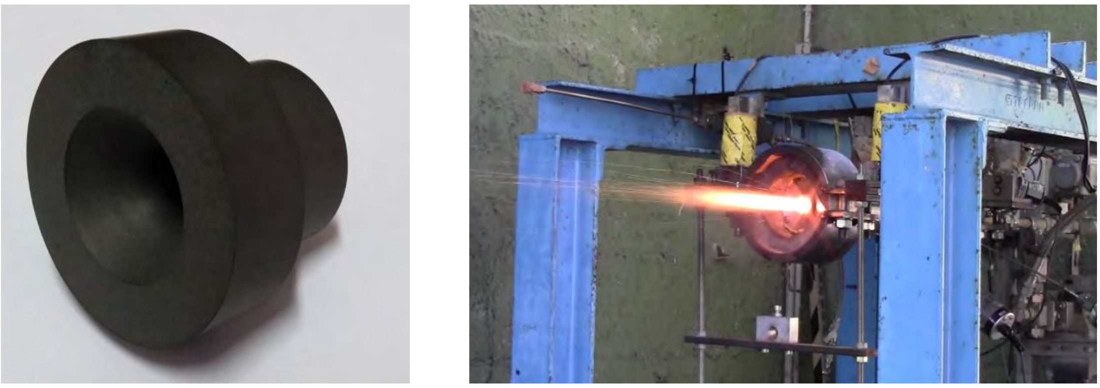

Fig. 2 UHT-CMC nozzle (left) and picture recorded during test in hybrid rocket laboratory (right).

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Tuesday 2019-09-24

Parallel room E1+E2

Topic 11 – Thermomechanical behavior and performance

On Performing Digital Image Correlation at Very High Temperatures

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Introduction

In the context of increasing the performance of future generations of aircraft engines, Safran Ceramics is developing for the hot zones of engines (high-pressure turbine applications) a new material composed of a 3D woven preform and a ceramic matrix obtained by liquid or gaseous process. In these applications, ceramic matrix composites (CMCs) are subjected to intense and multiaxial mechanical and thermal stresses possibly leading to damage that needs to be understood and controlled. To this end, multi-instrumented experiments (with infrared and visible light cameras) on CMC technological specimens were carried out, during which the material experiences severe 3D thermal loading (T_{max} > 1200°C). In such high-temperature environments, performing Digital Image Correlation (DIC) to measure accurate 2D or 3D surface displacements is challenging. Elevated temperatures affect both images (alteration of speckle pattern, changes and loss of contrast, variation of brightness during the experiment [1][2]) and measurement accuracy (heat haze effects increase uncertainties due to spurious displacements [2][3][4]). Therefore, a new spatiotemporal approach for Digital Image Correlation was proposed in order to filter out heat haze effects and correct the temporal variations of brightness and contrast.

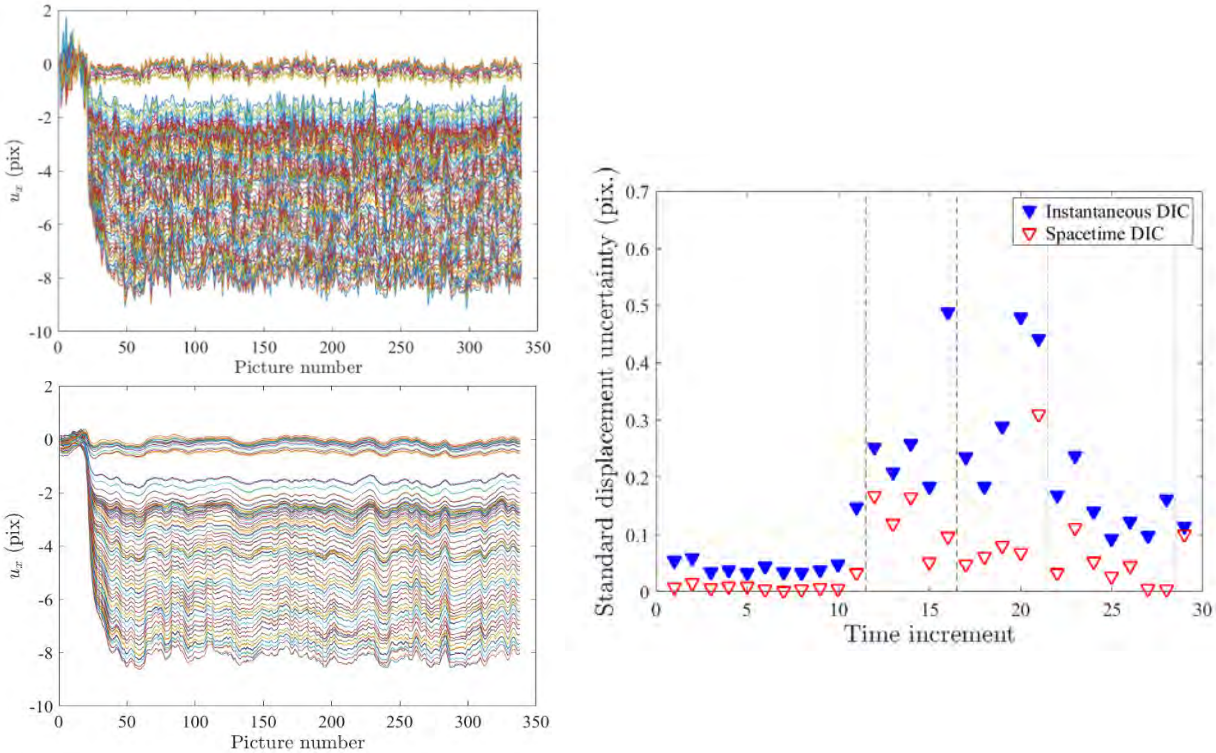
Results

Global spacetime registrations consist in measuring the spacetime displacement field $\boldsymbol{u}(\boldsymbol{x},t)$ over an image series $f(\boldsymbol{x},t)$ that minimises the overall grey level residuals between a reference image $\hat{f}(\boldsymbol{x})$ ($\boldsymbol{x}$ is any pixel of the Region Of Interest (ROI)) and the series of deformed images corrected by the displacement field $f(\boldsymbol{x}+\boldsymbol{u}(\boldsymbol{x},t),t)$. The displacement field is parameterised as a dyadic product between space and time shape functions (continuity in space – as obtained with global instantaneous DIC procedure [5], and time [6])

$$\boldsymbol{u}(\boldsymbol{x},t) = \sum_{i=1}^{n_x}\sum_{j=1}^{n_t} a_{ij}\boldsymbol{\theta}_i(\boldsymbol{x})\varphi_j(t)$$

Spacetime DIC then consists in finding the amplitudes a_{ij} of the sought displacement field solution of the spatiotemporal analysis. Two algorithms were developed for this purpose, namely, the temporal shape functions are either chosen *a priori* [7] or constructed through a modal analysis (with a Proper Generalised Decomposition) of the spacetime residuals [8]. Moreover, the grey level conservation assumption can be relaxed in the formulation of the registration problem through the addition of two corrections (brightness field $b(\boldsymbol{x})$ and contrast field $c(\boldsymbol{x})$) applied to the reference image $\hat{f}(\boldsymbol{x})$ [9], leading to a corrected reference image $\tilde{f}(\boldsymbol{x}) = \big(1+c(\boldsymbol{x})\big)\hat{f}(\boldsymbol{x}) + b(\boldsymbol{x})$).

These procedures were used to measure 2D displacement fields of CMC samples heated either by a flame (case 1), or a laser beam (case 2). For the first experiment (case 1), a CMC sample was heated by a flame and monitored by a visible light camera focusing on the edge of the sample. The image acquisition began at room temperature, then the sample was heated up to 1300°C. The spacetime (PGD) DIC procedure [8] leads to consistent displacements (elongation and deflection of the sample) compared to instantaneous DIC measurements, as highlighted in Figure 1(a). In addition, the approach removed an important part of convection effects, giving less noisy displacements over time. For the second experiment (case 2 [7]), a CMC sample was heated by a CO_2 laser beam at different temperature levels (650°C – 1050°C and over 1200°C) and monitored (among others) by a visible light camera focusing on the edge of the sample. The image acquisition also began at room temperature, then the sample was heated according to the previous thermal path, without and with a fan (to lower heat haze effects). The change of the standard displacement uncertainty is reported in Figure 1(b). Spacetime DIC reduced uncertainties at room temperature by a factor of 5 to 6. The quantitative effect of the addition of a fan was also investigated (reduction of temporal fluctuations by 40% for vertical displacements) and the use of the spacetime DIC approach led to even lower levels of standard uncertainties during the heating.



(a) (b)

Fig. 1 (a) Comparison of nodal horizontal displacements of CMC (case 1) with instantaneous DIC and spacetime DIC. (b) Temporal fluctuations of vertical displacements (case 2) with instantaneous and spacetime DIC for images acquired at room temperature (1 to 11) and during heating phases without (12 to 21) and with fan (22 to end).

Conclusion

The effect of elevated temperatures was investigated and spatiotemporal procedures with brightness and contrast corrections were proposed to overcome these challenges and measure actual displacements of various samples. These regularised measurements can now be used for identification purposes with significantly smaller uncertainties.

This work was supported under PRC MECACOMP, a French research project co-funded by DGAC and SAFRAN Group, managed by SAFRAN Group and involving SAFRAN Group, ONERA and CNRS.

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Fatigue Characterization of Melt Infiltrated SiC/SiC Ceramic Matrix Composites under Burner rig Conditions

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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.

Characterization of the damage process of C/SiC ceramic matrix composites by various monitoring

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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.

The influence of pyrolytic carbon layer on the bending properties of C/SiC composites prepared by LSI method

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Abstract: This paper investigated the influence of pyrolytic carbon (PyC) layer applied to the carbon fiber surface on the bending properties of three dimensional (3D) needled C/SiC composites prepared by liquid silicon infiltration (LSI) method. The PyC layer was fabricated by chemical vapor deposition (CVD) method, and several PyC layers with different thickness from 1um to 10um and different interfacial bonding characteristic were prepared. The microstructure and three-point bending test were studied, and the fracture behavior of C/SiC composites was discussed. The results showed that the PyC layer could effectively prevent the reaction between liquid silicon and carbon fiber during the LSI process when the PyC layer thickness was greater than 4~5um. And the bending strength of C/SiC composite increased with the increase of PyC layer thickness. The interface bonding strength between PyC and carbon fiber was another import factor affecting the flexural strength of C/SiC composite. The interface bonding performance could be adjusted by changing the CVD process parameters. The C/SiC composite reached the highest flexural strength of 380MPa and the flexural strain was about 1.0% when the interface bond strength was at a moderate level.

Keywords: Pyrolytic carbon layer, C/SiC, Liquid silicon infiltration, bending strength

Damage Evolution in an EBC-SiC/SiC System Due to Laser Heating Induced Thermal Gradients and Mechanical Loading

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L.Zawada - Universal Technology Corporation
J.Pitz - UES, Inc.
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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 to one face of dogbone test specimens, with and without the EBC, while under uniaxial tensile conditions using the fiber laser. 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 thermo-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 differently 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.

Tuesday 2019-09-24

Parallel room E1+E2

Topic 2 – Fibers & preforms

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

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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 $SiO_xC_yN_z$ phase are formed first, and then Si_3N_4 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, Si_3N_4 crystallites, C phase and amorphous $SiO_xC_yN_z$ phase with many defects and heterogeneous interfaces.

KEY WORDS： Si-C-N nanofibers, PDC, electrospinning, microstructure, crystallization

Tuesday 2019-09-24

Parallel room D1+D2

Topic 14 – CMC in Space Applications

MTG Star Tracker Assembly Bracket – A Successful Story of Fabrication light-weighted Structures

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ABSTRACT

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.

Keywords: Ceramic, Light Weighted Structure, HB-Cesic®, Silicon Carbide, SiC, Star Tracker, Ceramic Structure

FINE STRUCTURE CARBON-CARBON FOR APPLICATION IN SATELLITE ANTENNA REFLECTORS

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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 μm to 60 μm). Use of the carbon-carbon for the reflector antenna manufacturing allows to minimize the roughness of its surface up to R_a 0.10-0.15 at a coefficient of thermal expansion around $0.5 \cdot 10^{-6}$ K^{-1}. The characteristics of the reflector made from the CMC are demonstrated.

VINCI Engine Composite Nozzle Extension For Ariane 6

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ABSTRACT

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 hardwares 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.

Black Engine CMC Space Propulsion Technology

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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.

CMC based Structures for Hypersonic Flight

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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.

Characterization and modeling of bending properties of continuous fiber reinforced C/C-SiC sandwich structure

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Abstract

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.

Keywords: ceramic matrix composite; sandwich structure; C/C-SiC; bending properties; characterization; simulation

Tuesday 2019-09-24

Parallel room D1+D2

Topic 16 – Aeronautic Applications

Ceramic Matrix Composites Taking Flight at GE Aviation

Jim Steibel and Brian Dix
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Abstract

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.

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

Luchao Sun and Jingyang Wang

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 °C and molten CMAS (calcium-magnesium alumino-silicates) corrosion at 1500 °C 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.

Keywords: Directionally Solidified Eutectic; Mechanical Properties; Hot Gas Corrosion; Calcium–Magnesium–Aluminosilicate (CMAS)

Wednesday 2019-09-25

Amphi B

Plenary

Mechanical Behavior of an Oxide-Oxide Ceramic Matrix Composite at Elevated Temperature in Air and in Steam[1]

M.B. Ruggles-Wrenn

Air Force Institute of Technology, Wright-Patterson Air Force Base, OH 45433, USA

Abstract

Mechanical behavior of an oxide-oxide ceramic composite was investigated at 1000-1200°C in air and in steam. Composite consists of a porous alumina matrix reinforced with mullite/alumina (Nextel™720) fibers, has no interface between fibers and matrix, and relies on the porous matrix for flaw tolerance. Tensile and compressive stress-strain behaviors were examined and tensile and compressive properties measured. Tensile creep and compressive creep behaviors were investigated. Steam accelerated creep rates and dramatically reduced creep lifetimes. Tension-tension fatigue behavior was evaluated at 0.1, 1.0 and 10 Hz. The composite exhibited excellent fatigue resistance in air. Steam significantly degraded the tension-tension fatigue performance. Tension-compression fatigue behavior was studied at 1.0 Hz for ratio of minimum to maximum stress of -1.0. Tension-compression fatigue was considerably more damaging than tension-tension cycling. Steam reduced tension-compression cyclic lives by an order of magnitude. Retained properties of all specimens that achieved creep or fatigue run-out were characterized. Composite microstructure, as well as damage and failure mechanisms were investigated.

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[1] The views expressed are those of the authors and do not reflect the official policy or position of the United States Air Force, Department of Defense or the U. S. Government.

Wednesday 2019-09-25

Amphi B

Topic 8 – EBC/TBC

TOWARD THE UNDERSTANDING OF FAILURE MECHANISMS OF ENVIRONMENTAL BARRIER COATINGS

Manon FERNANDEZ[1,2], Guillaume COUEGNAT[1], Francis REBILLAT[1]

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In the aeronautic field, current issues are the reduction of noise pollution, consumption and emissions of NO_x and CO_2. In this context, structures weight and operating temperature of the engines are key parameters. Among the candidate materials for such applications, SiC based ceramic matrix composites (CMC) have been selected because of their excellent high temperature properties, low density and good thermomechanical stability. In use, the composite is subjected to severe thermomechanical and physicochemical conditions that can lead to the degradation of the material and to a limitation of its lifetime [1].

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 (EBC for environmental barrier coating) as well as decreasing their surface temperature (TBC for thermal barrier coating). 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; the composite is directly exposed to the environment. 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. Morphological observations of these systems after aging were performed and their adhesion was quantified [2].

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 under high gas flow 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. This modeling considers the silica growth at the bond coat/EBC interface, roughness and constituent's properties as a function of temperature.

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Thermal fatigue cracks on environmental and thermal barrier coating under thermal gradients

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2: SAFRAN, Safran Ceramics, France
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Introduction

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 [1]. It is therefore essential to protect the CMC 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 multi-axial thermal gradients but also mechanical loading. This makes standard tests used for characterization ill-adapted to assess damage initiation and growth or estimate lifetime [2]. For that reason, the coated CMC is locally heated using a high power (3kW) high heat flux CO2 laser to perform thermal fatigue tests.

Results

As illustrated on Figure 1, 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 [3] thanks to a speckle pattern created using SiC powders and optical filters. The through-thickness thermal gradient can be controlled by active air-cooling at the back side of the sample and a bichromatic pyrometers. Damage onset and growth is detected in-situ using acoustic emission (AE) with wire waveguides.

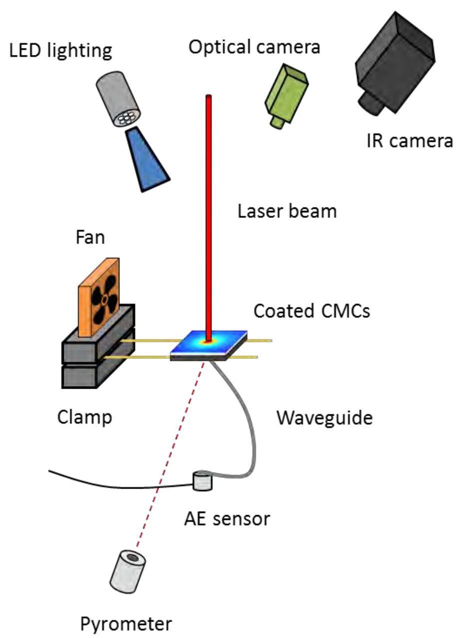
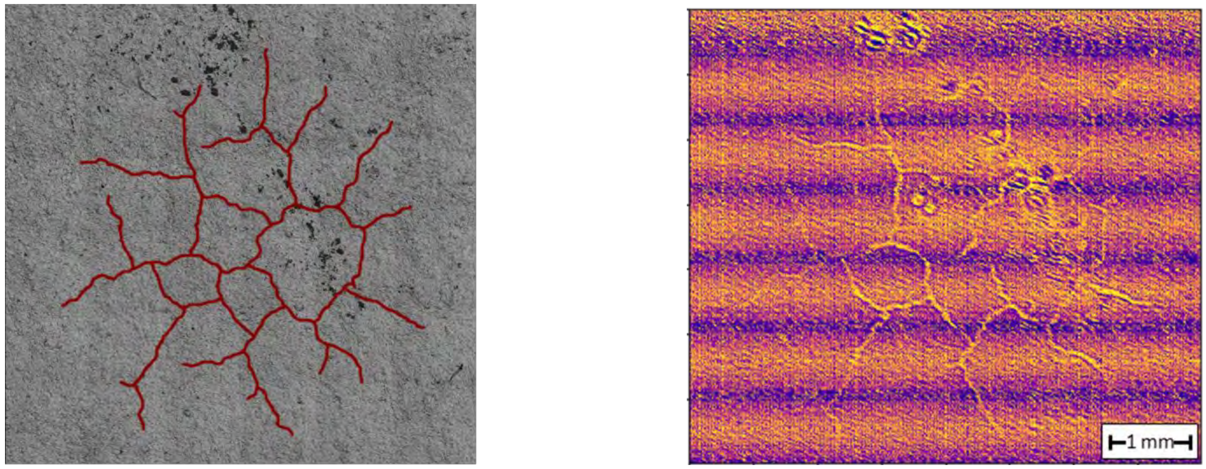


Figure 1: Multi instrumentation of very high temperature test

One hundred cycles with a maximum temperature of 1300°C were performed on the sample. After the test, non-destructive evaluation using laser spot thermography [4] reveals the development of thermal fatigue micro cracks (~1µm opening) located under the laser beam, SEM observations confirmed these results as shown on Figure 2.



(a) (b)

Figure 1: Development of crack network under thermal fatigue test (a) SEM observation (b) laser spot thermography observation

Conclusion

Thermal fatigue test performed on a coated ceramic matric composite shows the importance of thermal gradients which induce cracking in the coating. Non-destructive evaluation using laser spot thermography has been used to observe these micro cracks. The influence of this damage on the lifetime of the system is to be studied.

This work was supported under PRC MECACOMP, a French research project co-funded by DGAC and SAFRAN Group, managed by SAFRAN Group and involving SAFRAN Group, ONERA and CNRS.

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

OPILA Elizabeth
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.

CMAS degradation of environmental barrier coatings in presence of water vapor and air

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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 $33CaO$-$9MgO$-$13Al_2O_3$-$45SiO_2$ 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 ZrO_2 und YAG the penetration and reaction of CMAS was less compared to the rare-earth silicates (Y_2SiO_5, Yb_2SiO_5 and $Yb_2Si_2O_7$). 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 SiO_2 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 Yb_2SiO_5 or $Yb_2Si_2O_7$ 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 $Yb_2Si_2O_7$ layer was significantly higher than that of the Y_2SiO_5 layer.

Corrosive Properties of RE-doped Silicate for Environmental Barrier Coatings

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Seongwon Kim[3], Yoon-Suk Oh[3], Sung-Min Lee[3] and Hyung-Tae Kim[3]

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Keywords: Y_2SiO_5, Yb_2SiO_5, Environmental barrier coatings, CMAS, Volcanic ash

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/SiC_f, Si_3N_4 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 Si_3N_4 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 Y_2SiO_5 EBCs (environmental barrier coatings) on SiC was investigated. The hot corrosion between Y_2SiO_5 coatings and artificial CMAS ($CaO-MgO-Al_2O_3-SiO_2$) 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 Y_2SiO_5 coatings by nano indentation method. The isothermal heat treatment improves the hardness and Young's modulus of Y_2SiO_5 coatings. In addition, high-temperature corrosion behavior of volcanic ash and CMAS on sintered Yb_2SiO_5 are investigated.

Wednesday 2019-09-25

Parallel room H1+H2

Topic 9 – PDCS

Wednesday 2019-09-25

Parallel room F1+F2

Topic 1 – Modeling

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

Sébastien Denneulin, Thomas Revel, Thomas Vandellos, Jérémy Blachier

SAFRAN Ceramics, France

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.

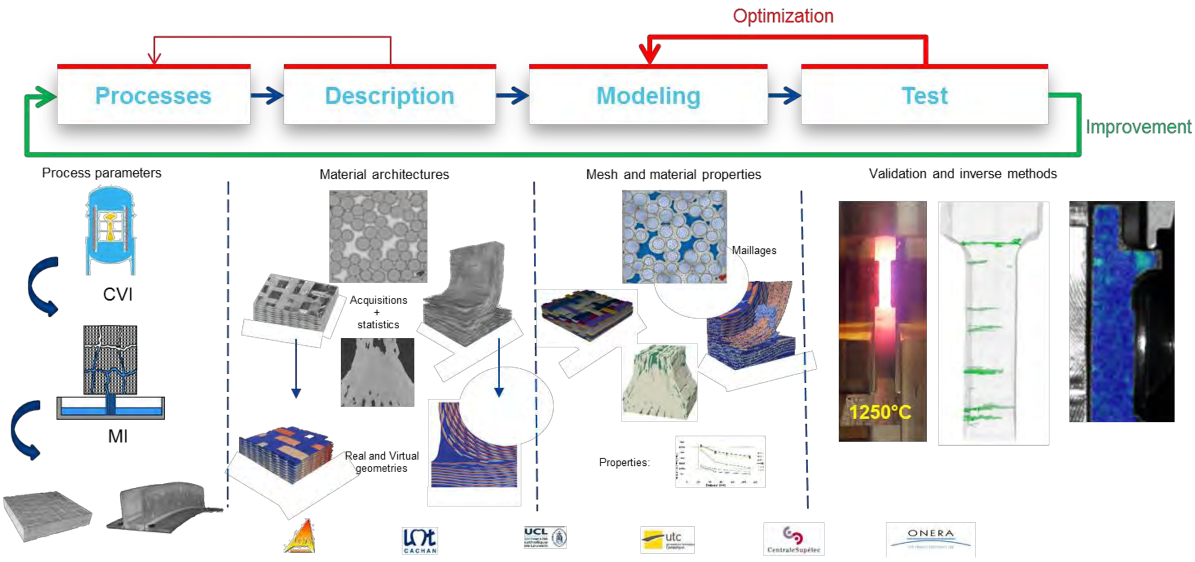

Fig. 2. Multi-scale studies framework

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].

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Wednesday 2019-09-25

Parallel room E1+E2

Topic 2 – Fibers & preforms

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

ChuChu Guo, Laifei Cheng, Fang Ye

Abstract: 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 μm. As the pyrolysis temperature increases from 1050 ℃ to 1550 ℃, 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 ℃ 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.

Keywords: SiC nanowires, vapor-liquid-solid growth mechanism, pyrolysis temperature, precursor state

Wednesday 2019-09-25

Parallel room E1+E2

Topic 4 – Oxide/Oxide CMC S

New Developments in Oxide-Oxide Ceramic Matrix Composites Manufacturing

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M. Simpson
3M Company
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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.

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

Itsaso Echeverria, National Composites Centre, UK / University of Surrey, UK
Dr. David Fishpool, National Composites Centre, UK
Dr. Virtudes Rubio, National Composites Centre, UK
Prof. Robert Dorey, University of Surrey, UK
Prof. Julie Yeomans, University of Surrey, UK

Abstract

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 from 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.

Wednesday 2019-09-25

Parallel room D1+D2

Topic 16 – Aeronautic Applications

Advances in developing an alternative to ceramic matrix composites in high temperature applications

S.T. Mileiko
Institute of Solid State Physics of Russian Academy of Sciences

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 (Mo_3Si and Mo_5SiB_2) 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. A result is illustrated by X-Ray spectra presented in Fig. 1. One can see the phases mentioned above.

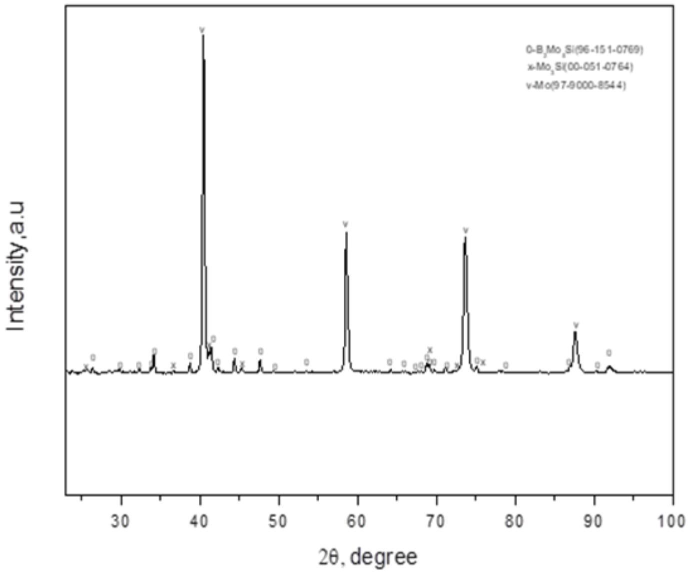

Fig. 1. Typical X-Ray spectra obtained on a molybdenum-based matrix.

Composites with various oxide fibres and $Mo+Mo_3Si+Mo_5SiB_2$ matrix have strength characteristics higher than simple oxide/Mo composites (OMCs). Figure 2Fig. 2 illustrates an effect of the change in the matrix composition at the strength of composites reinforced with $YAlO_3$-based fibres. The values of strength at 1400ºC of the composites under the development and simple OMCs, both are reinforced with the fibres crystallised at a rate of ~ 50 mm/min, are 370±43 and 257±29, respectively. Critical stress intensity factor of the composites is between 15 and 20 MPa·m$^{1/2}$ as compared with that values < 10 MPa·m$^{1/2}$ for the strongest $Mo_{ss}+Mo_3Si+Mo_5SiB_2$ alloys and ~ 30 MPa·m$^{1/2}$ for simple OMCs. High fracture toughness of the composites is also revealed by a view of the notched specimens after testing, Fig. 3.

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.

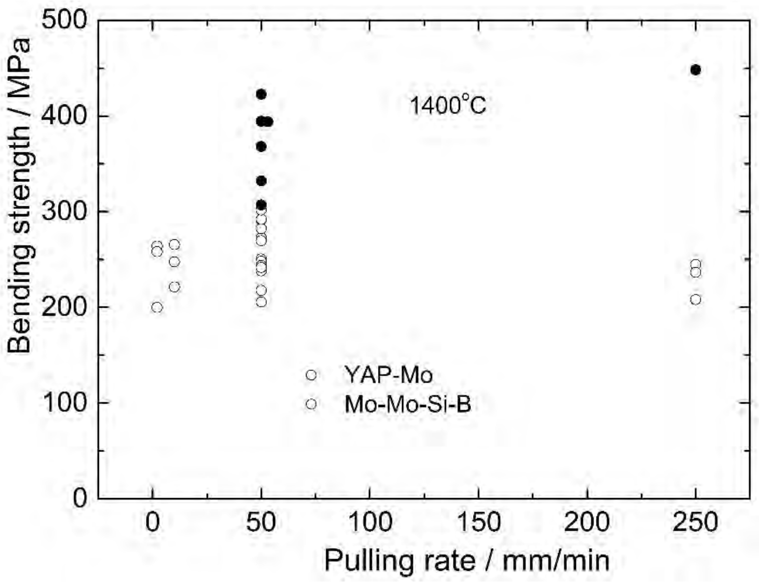

Fig. 2. High temperature strength of the composites with the pure molybdenum matrix and matrix containing ceramic particles versus crystallization rate of the reinforcing fibres ($YAlO_3$).

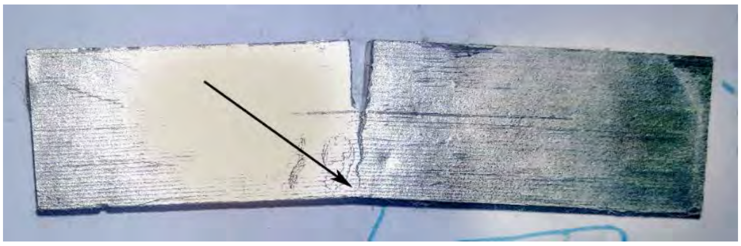

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Fig. 3. The notched specimens after testing. The distance between supports is 60 mm, the specimen thickness is ~ 5 mm.

To conclude, it should be said that reinforcing molybdenum matrix filled with ceramic particles with oxide fibres crystallised from the melt allows producing fibrous composites for very severe service conditions.

***

The work is supported by Russian Science Foundation, project 16-19-10624. Also, the author is thankful to the active members of his team, namely Drs A.A. Kolchin, V.M. Prokopenko, P.M. Bazhin, E.L. Vardanyan, S.N. Galyshev, B. Miktybekov, and research engineers N.A. Prokopenko, O.F. Shachlevich, A.Ya. Mizkevich, V.A. Chumichev, S.A. Abashkin.

Development and evaluation of the fracture toughness of SiC-based ceramic matrix composite

By: Justine Delage

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Abstract:

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.

Thursday 2019-09-26

Amphi B

Plenary

Production and Development of Carbon/Carbon Composites at Schunk Kohlenstofftechnik

Roland Weiss
Schunk Group (retired), Heuchelheim, Germany

Abstract

As known, carbon/carbon composites were originally developed for space and military applications. The carbon/carbons can be more or less tailored according the specific requirements of the final components. Modifications are feasible not only by different processing routes and reinforcement patterns, but also by various post treatments. The possibilities enable a family of carbon/carbon composites which will be shown in more detail.

The industrial use of Carbon/carbons and the necessary tailoring of properties will be shown for some typical industrial application fields. All industrial high temperature applications of CMC components are cost driven. Therefore, a continuous progress in manufacturing technologies and design of component is necessary. The CMC materials as well as the manufacturing process have to be cost efficient and tailored in order to fulfill mechanical, thermal and corrosion requirements.

The talk will show some examples from the early beginning of carbon/carbon developments at Schunk until the state of art for industrial carbon/carbon manufacturing.

Carbon/carbons have the potential to improve energy efficiency for high temperature processes and thereby to reduce the CO2 foot print for the process as well as for the resulting products. Such energy and cost savings will be demonstrated by new furnace and relining concepts based on carbon/carbons.

Thursday 2019-09-26

Amphi B

Topic 8 – EBC/TBC

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

Authors

Keywords: C/C-SiC composite, LSI, injection moulding, large-scale production, pyrolysis process

Nier, N.; Ahmad, H.; Gurk, H.; Päßler, E.; Roder, K.; Nestler, D.; Kroll, L.; Wagner, G.

Abstract

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.

Thermal properties and crystal structure of ytterbium titanate

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Abstract

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 $Yb_2Ti_2O_7$ (high *n*)/$Yb_2Si_2O_7$ (low *n*) and Yb_2TiO_5 (high *n*)/Yb_2SiO_5 (low *n*) are expected to be potential candidates that also exhibit excellent corrosion resistance. Furthermore, $Yb_2Ti_2O_7$ has lower thermal conductivity than conventional yttria-stabilized zirconia. The pyrochlore $Yb_2Ti_2O_7$ is transformed to the fluorite Yb_2TiO_5 with an increase in the content of Ti in the $Yb_{2+x}Ti_{2-x}O_{7-x/2}$ 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 $Yb_{2+x}Ti_{2-x}O_{7-x/2}$ 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.

Pyrochlore lattice tuning towards extremely low thermal conductivity for potential thermal barrier coating applications

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Abstract

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.

Development of Environmental Barrier Coatings for SiC/SiC Ceramic Matrix Composites via CVD

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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.

Effect of difference in material of substrate on aerosol deposited mullite coating under heat exposure

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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.

Environmental Barrier Coatings Made by Different Thermal Spray Technologies

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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.

Thursday 2019-09-26

Parallel room H1+H2

Topic 17 – Energy Applications

$TiNb_2O_7$/keratin-derived carbon microtubes composites as anode materials for lithium-ion batteries

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$TiNb_2O_7$ (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, $TiNb_2O_7$ crystalline phase is formed with specific surface area (SSA) of 28 m^2g^{-1}, whereas TNO/CMT yields $TiNb_2O_7$ and non-stoichiometric $(Ti_{0.712}Nb_{0.288})$ O_2 as crystalline phases with SSA of 89 m^2g^{-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.

FOCUS ON THE THERMAL BEHAVIOR OF SILICON CARBIDE COMPOSITES AS ACCIDENT TOLERANT FUEL CLADDING

Thermal characterization developments and progress

(Proposal abstract for topic 17 "Advanced materials for sustainable energy")

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Abstract 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_f/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_f/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_f/SiC composites and the evolution of their thermal behavior regarding both the LWR environments and ATF requirements.

Keywords Silicon carbide, SiC_f/SiC composites, Accident Tolerant Fuel, Nuclear, Thermal characterization.

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ENHANCING ACCIDENT TOLERANCE OF NUCLEAR FUEL WITH SIC-BASED CLADDING

Benefits in accidental conditions, challenges and ongoing developments

(Proposal abstract for topic 17 "Advanced materials for sustainable energy")

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Abstract Following Fukushima-Daiichi nuclear disaster, French Nuclear Institute was led to consider silicon carbide based continuous fiber ceramic matrix composite materials (SiC_f/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 SiC_f/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.

Keywords silicon carbide, SiC_f/SiC composites, Accident Tolerant Fuel, Nuclear.

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**Accident-tolerant SiC/SiC composite fuel cladding materials –
the H2020 IL TROVATORE approach to performance optimization**

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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°C). 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°C (β-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 (Fig. 1). 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°C), and assesses the radiation tolerance of various SiC/SiC composites using ion/proton irradiation.

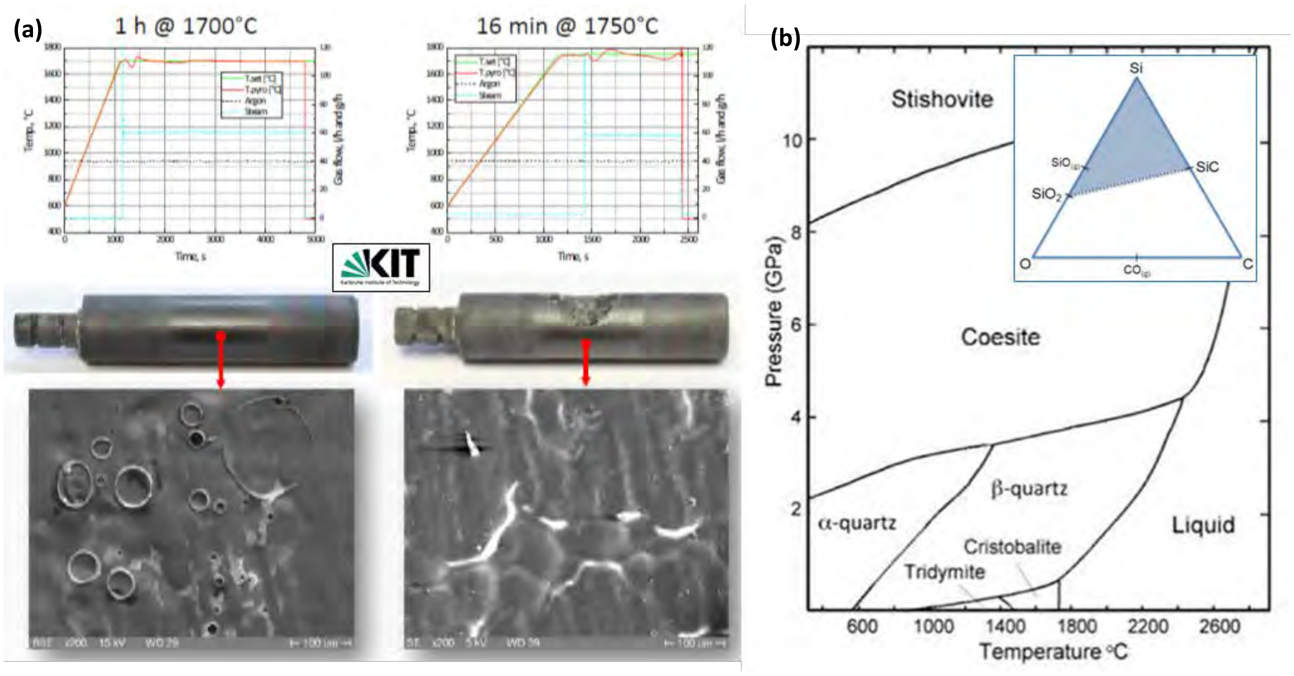

Fig. 1. (a) Isothermal steam tests on SiC/SiC; (b) silica phase diagram (inset: the SiO_2-SiC equilibrium is relevant in accidental conditions).

OVERVIEW OF SIC/SIC DEVELOPMENTS FOR CLADDING APPLICATION IN GAS FAST REACTOR

(Proposal abstract for topic 17 "Advanced materials for sustainable energy")

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Abstract 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.

Keywords silicon carbide, SiC/SiC composites, GFR, Nuclear.

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DEVELOPMENT OF SILICON CARBIDE AS A NUCLEAR FUEL CLADDING

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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.

This material is based upon work supported by the Department of Energy under Award Number DE-NE0008824.

Thursday 2019-09-26

Parallel room F1+F2

Topic 1 – Modeling

SiC/SiC composites crack modeling at the fiber scale using a phase-field approach

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Abstract — SiC/SiC Ceramic Matrix Composites designed for plane engines have rich microstructures generated through successive manufacturing processes. Furthermore they are subjected to various degradation mechanisms due to thermomechanical loads. As a consequence the finite element modeling of such material is of great interest in the engineering field. In order to help the understanding of the non-linear phenomena within the fiber-scale microstructure and optimize the manufacturing processes, an implementation of a diffusive crack model in a commercial code is suggested in the present work. New numerical investigations will be conducted: fiber scale 3D microstructure damaging due to post-manufacturing cooling and a simulation of a crack deflection along a fiber submitted to a tensile stress will be presented.

Keywords — phase-field modeling, fiber-scale microstructure, brittle fracture, longitudinal crack deflection.

I) Introduction

SiC/SiC ceramic matrix composites developed for civil aviation have to replace some metallic parts in the hot sections of aircraft engines such as the Safran LEAP. 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 [7], 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 complex, which makes its finite element modeling a challenge. To develop a tool that helps to understand the non-linear behavior 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 diffusive crack model based on a gradient-type damage model using a commercial code. Various formulations can be found from the literature but 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 (i) initiation, (ii) propagation and (iii) deflection of cracks without mesh size dependency if the elements are small enough. New numerical investigations will be conducted: 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.

II) State of the art of crack modeling

Modeling crack initiation, propagation and deflection is of great interest for engineering applications. Numerous studies have been conducted in order to describe brittle fracture mechanisms within SiC/SiC composites, the current section draws a short state of the art of the different existing methods adapted to ceramic matrix composites.

A first idea consists in initiating and propagating cracks using a stress criterion for the different phases of a fiber-scale microstructure [9]. Crack initiation is located on the microstructure defects and the propagation is ensured by duplicating the nodes of the 2D model so an explicit crack is created. Nonetheless such strategy is highly mesh dependent and 3D crack surfaces seem non-reasonable to describe the microstructure. Cohesive elements associated with a damage law are able to initiate and propagate a crack within the microstructure. If damage mechanisms are known cohesive elements can be located for instance to model fiber-matrix debonding [5], else cohesive elements can be introduced in the whole structure [11]. The limitations lie in the mesh dependancy and the elastic energy influx which contributes to decrease the condition number of the structure stiffness matrix. Enriching the Galerkin formulation of the approached displacement field is an alternative strategy for crack modeling. Based on the partition of unity method, the extended finite element method (XFEM) enables the addition of degrees of freedom associated with levelset functions to describe split elements by the crack and extra degrees of freedom associated with analytical crack tip enrichment functions. The XFEM is free from mesh conforming but is limited by the quality of the analytical enrichment functions which are only well-known in infinite domains [3]. Moreover crack initiation is impossible, methods like Continuum Damage Models [10] must be defined to take into account cracks appearing.

All the three previous strategies present limitations when surface cracks in three dimensions problems must be described. To overcome these defects it is easier to spread the damage field around the sharp crack using a length scale defined as a phase-field that interpolates between the unbroken and the broken state of the material. Such method is based on a variational approach introducing a gradient-type dissipative function. The obtained damage law is able to initiate and propagate a crack regarding to the irreversibility of the crack propagation. Furthermore the method converges on Griffith model [4], it is able to treat crack duplication [6] and crack deflection within a composite [2]. The length scale of the phase-field sets the mesh size [8].

In this contribution it is chosen to use a phase-field approach regarding to the robustness of the method and its mechanical content while avoiding mesh dependency. Moreover among the different formulations summarized in [1], it can be found a heat equation-like damage law which is convenient for a commercial code implementation.

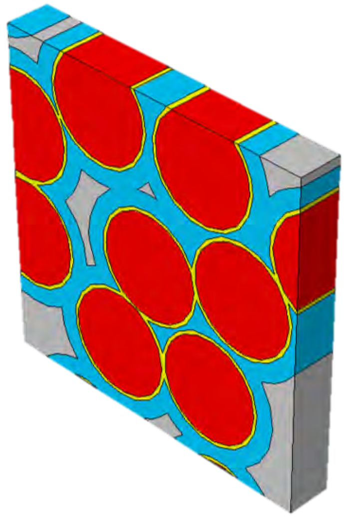
III) Implementation of the phase-field model in a commercial code

A specific damage law is chosen for this paper from [8] and is written below:

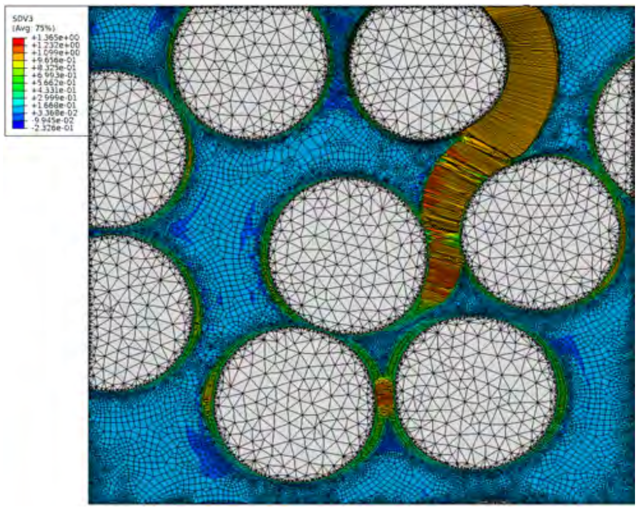
$$\boxed{\eta\dot{d} - G_c l \Delta d = 2\mathcal{H}(\boldsymbol{x},t) - \left(2\mathcal{H}(\boldsymbol{x},t) + \frac{G_c}{l}\right) d} \tag{1}$$

$\eta\dot{d}$ is a numerical viscous over-force, G_c is the critical energy release rate of the material and l is the length-scale parameter. $\mathcal{H}(\boldsymbol{x},t)$ is defined as a driving force that governs the evolution of the damage d. $\boldsymbol{x}$ is the position of a point in the domain and t is the current time.

The aim is to integrate such heat equation-like law into Abaqus while taking into account the thermal effects on the microstructure. For this purpose a couple (η, l) must be fixed regarding the SiC/SiC composite microstructure. In practice the driving force is defined as the maximum elastic energy due to the contribution of positive principal strains reflecting the tensile solicitations that contribute to crack



(a) Reference Elementary Cell used for the study



(b) Crack profile obtained after a tensile test on the CER

Figure 1: Reference Elementary Cell including different SiC/SiC composites phases except porosities. Scalar damage field is also obtained using Abaqus routines

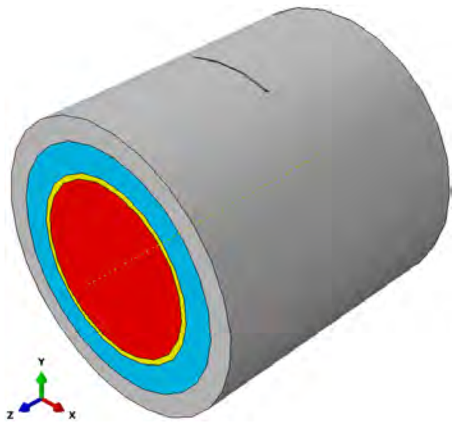


Figure 2: Test structure in order to simulate crack deflection along a fiber. A notch localizes the crack initiation.

opening. Within the framework of a quasi-static crack propagation, the use a viscous parameter is justified by the necessity to avoid the snap-back phenomenon highlighted in [12]. The length-scale l has to be chosen with the help of the features in the microstructure. The issue of taking into account the temperature effects is also raised due to the use of the thermal routine to solve EQ (1). A hypothesis of independence of the temperature from the mechanical quantities will be formulated in order to solve in a first time the thermal problem before solving the mechanical one.

Research will be conducted using FIGURE 1(a) as a support, investigations will lead to crack patterns like FIGURE 1(b). A transverse crack deflection along a fiber will also be shown with the help of an elementary geometry like shown in FIGURE 2.

Acknowledgements

The authors wish to acknowledge the support of Safran Ceramics for funding this research.

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

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Ceramic Matrix Composites are light materials with excellent mechanical properties at high temperature, which make them potential candidates for applications in hot engine parts, thus contributing to aircraft weight reduction. Specifically, 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 physico-chemical reaction with the environment over a large range of temperature. There is no dependence of the behaviour on temperature and no viscosity below 1000°C, 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, where the number of flying cycles is high. In order to use these materials, it is necessary to develop efficient calculation strategies for the design of composite parts submitted to both static and fatigue loadings. The aim of this study is 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.
Despite a large number of studies on the behaviour of oxide/oxide composites in the literature, there is, to our knowledge, no damage model able to determine the life of composites structures submitted to spectral fatigue loadings, representative of real industrial loadings.
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 static part of the model is based on Ben Ramdane's thesis work [1]. The extension of the model to fatigue loading is carried out using a kinetic damage law initially proposed

for organic matrix composites [2], and adapted to the specificities of oxide/oxide composites. The use of a kinetic formalism 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 (greater than 10^4), all cycles being simulated with this type of methodology.
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 [3] 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. Figure 1 shows a damage field of an open-hole plate oriented at 45° after 100,000 cycles. The simulation lasts 3h30 (with 4 CPUs) whereas it would require several weeks of calculation by simulating all cycles with the kinetic model. The error generated using the proposed cycle skip method is checked during the calculation. During the first thousand cycles, it remains low (<5% on damage values) compared to the reference solution obtained by simulating all cycles.

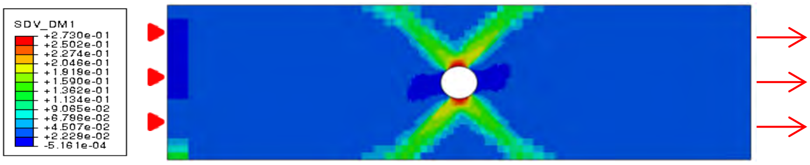

Fig. 1. Matrix damage field in the main composite direction of an open-hole plate oriented at 45° after 100,000 cycles under imposed force and R=0.05

This numerical methodology is used to simulate the evolution of the mechanical properties of an open-hole plate subjected to fatigue loading. The model's predictions, particularly in terms of loss of stiffness, will be compared with the results of an experimental campaign planned for early 2019 at Onera.

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

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September 22 - 26, 2019

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Context

During an atmospheric reentry, the heat shield protecting the body will undergo very high temperature (> 2000 K) and pressure (> 100 bars) on its surface. This is why thermal protection systems (TPS) are used, composed especially of **3D carbon/carbon composites**. Numerous phenomena will occur on the surface, in particular heterogeneous chemical reactions between the carbon and the surrounding air (oxidation, nitridation) and, at higher temperatures, sublimation; these reactions will cause the recession of the surface of the composite heat shield.
All along the reentry phase, the flow around the body will evolve, with in each regime characteristic surface roughness features caused by the ablation of the composite material. The laminar flow is associated with a microscopic roughness dimension [Levet(2017)], due to the material manufacturing history. Then the slight ondulations of the material will favour the **transition to turbulence** on located spots on the surface, and will give birth to macroscopic patterns with a centimetre scale (Fig. 1a). The flow will finally become **fully turbulent**, with the development of a new type of patterns of a millimetre scale [Hochrein et Wright Jr(1976)], no more localised this time but generalised to the whole surface (Fig. 1b). These characteristic patterns are known as **"scallops"**. They are able to multiply the heat transfer and the recession rate by more than 2 [Wool(1975)]; therefore understanding their formation and behaviour is a necessity for a better design of TPS.

Theoretical approach

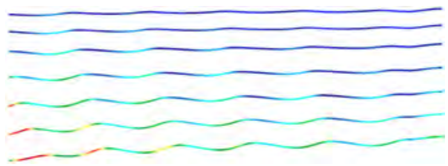
The stability of these morphological features is studied, considering the coupled viscous boundary-layer equations, the convection-diffusion of an oxidant, the Hamilton-Jacobi equation for the surface recession and the heterogeneous reaction on the surface. A model leading to the amplification of a surface perturbation is given, involving the **surface roughness** and the **pressure gradient** within the flow.
A linear stability analysis is carried out to determine the unstable regimes. The parameters upon which the stability depends are the Reynolds number (inertial/viscous effects), Damköhler number (reaction/diffusion), Schmidt number (viscous/diffusion) and surface roughness.

Numerical validation

A numerical approach is also carried out, in order to confirm the results of the theoretical work and evaluate the system beyond linearity. A strong coupling between the turbulent flow and the surface

(a) Transitional patterns (internal communication CEA)

(b) Turbulent patterns ([Budde(1975)])



(c) Evolution of the surface during the simulation

Figure 1: Different surface patterns according to flow regime

recession is performed using the open-source toolbox OpenFoam [Weller et al.(1998)] on a simplified case. The simulation is incompressible, and because the focus is set on the interaction between turbulence and surface recession, only one oxidizing species is taken into account, without thermal effects.
A Reynolds-Averaged Navier-Stokes (RANS) method is considered, with the two-equation turbulence model $k - \omega$ SST [Menter(1993)]. The flow is solved with a finite-volume scheme, and the evolution of the surface with a level-set method. To perform an efficient coupling between these two phenomena occurring at very different time scales, the PIMPLE algorithm is considered allowing Courant numbers much greater than 1. Numerical results confirm the existence and development of scallops (Fig. 1c). They arise apparently from the lag between the surface oscillations and the oscillations of the turbulent viscosity [Claudin et al.(2017)].

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Thursday 2019-09-26

Parallel room E1+E2

Topic 4 – Oxide/Oxide CMC S

Understanding the Slurry Infiltration and Retention Characteristics of Higher Denier Nextel™ Fabrics in Oxide-Oxide CMC Systems

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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 3M™ Nextel™ 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.

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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 Al_2O_3 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 %.

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

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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).

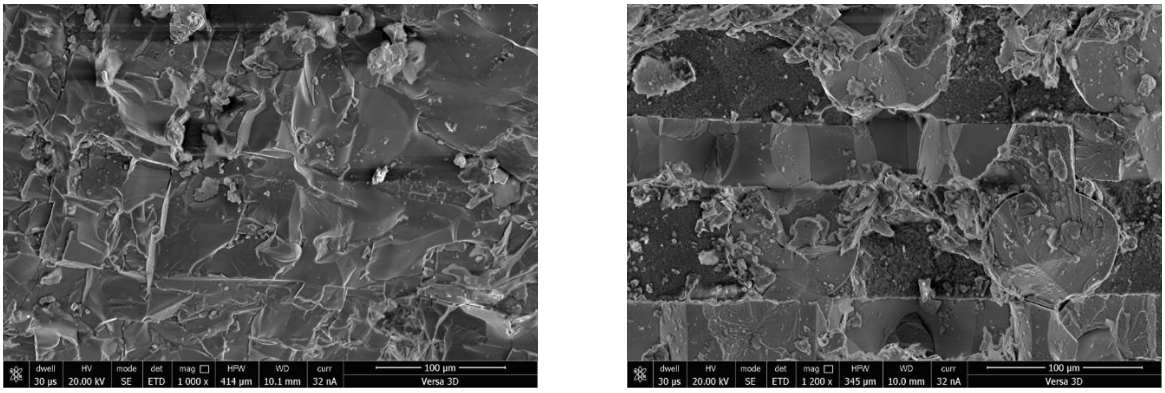
Two examples of the usage of oxide fibres in various matrices

M.Yu. Nikonovich, A.A. Kolchin, S.N. Galyshev, V.M. Prokopenko, S.T. Mileiko

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 Al_2O_3-$Al_5Y_3O_{12}$ eutectic. The task of protection against oxidation is solved quite simply. In order to evaluate the crack resistance, the first experiments on the composites were carried out, the fracture surface of one of the specimens is shown in Fig. 1.



a) b)

Fig. 1. The fracture surface of the composite of a hierarchical structure.

a) Oxide matrix; b) the composite

Preliminary experiments have shown a possibility of creating sufficiently crack resistant structures of this type with critical stress intensity factor reaching 25 MPa·m$^{1/2}$ (Table 1). This is surprising, whereas the fracture surface of the oxide-molybdenum composite is not typical for non-brittle fracture of the composite specimens.

Table 1. The values of the critical stress intensity factor and the strength of specimens with a notch.

Specimen number	K*	σ_N^*
	MPa·m $^{1/2}$	MPa
V1137	19.2	327
V1136	13.3	239
V1135	24.9	399

The second example is fibrous oxide composites without the matrix. In this case, there were 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. The fracture surface of the oxide specimens has a typical form of quasi-plastic fracture (Fig. 2). The highest strength (about 200 MPa) showed specimens obtained at the sintering temperature 1700°C. Preliminary test results indicate the effectiveness of such composites.

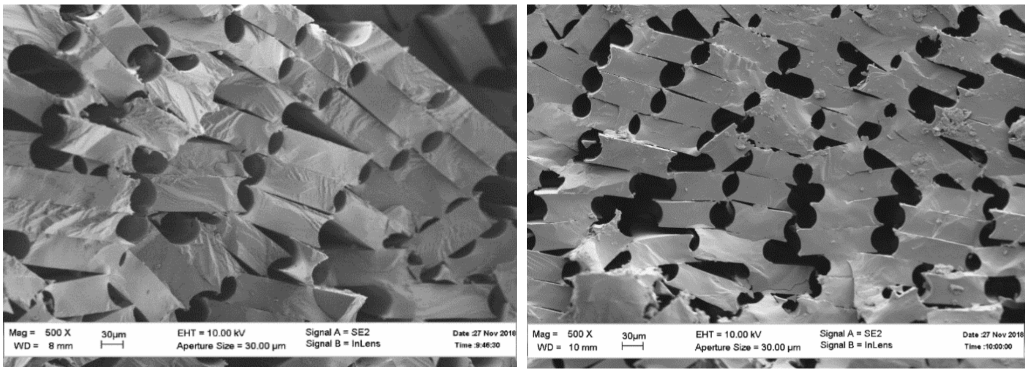


Fig. 2. The fracture surface of sapphire specimens at 1550 and 1700°C, on the left and right hand, respectively.

***

The work is supported by Russian Science Foundation, project 16-19-10624. Also, the authors are thankful to their colleagues N.A. Prokopenko, O.F. Shachlevich, A.Ya. Mizkevich, V.A. Chumichev, S.A. Abashkin.

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

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(1) CEA-LCTS; (2) Université de Bordeaux-LCTS; (3) CNRS-LCTS

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.

The authors want to greatly thank Mr Jean François Leon and Mr Samuel Couillaud from Galtenco Solution company, for the development of the new fast sintering technique.

Thursday 2019-09-26

Parallel room D1+D2

Topic 15 – Terrestrial Transport & Industrial Applications

Lightweight Metal-Ceramic Hybrid Brake Disc for Electric-Powered Vehicles: Concept and Prototype

Department of Ceramic Materials Engineering

T. Balzer, N. Langhof, W. Krenkel

Abstract

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.

Thursday 2019-09-26

Parallel room D1+D2

Topic 6 – Additive Manufacturing

Stereolithographic Additive Manufacturing of Sound Absorption Cavities

KIRIHARA Soshu
Osaka University

Complicated ceramics structures were designed by three-dimensional computer graphics and fabricated by using Stereolithographic processes. Photosensitive acrylic resin including with the ceramics particles were spread on a substrate by using a mechanical knife edge. An ultraviolet laser beam of 355 nm in wavelength was focused into 100 μm in diameter and scanned to draw a cross sectional solid pattern. A composite precursor was obtained successfully thorough continuous laminations. The ceramics component could be created by dewaxing and sintering. In this investigation, sound absorption structure with structural property of Helmholtz chamber were designed and fabricated by this process. Sound wave generated by thermal spraying was controlled by the Helmholtz structure. The frequency spectra were plotted by computational fluid dynamics and acoustic simulation. According to the theoretical procedure, a soundproof equipment for gas flame noise from thermal spraying were designed and fabricated by stereolithography.

Key Words: Stereolithography, Additive Manufacturing, Helmholtz chamber

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

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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 mainly granted by the Nouvelle Aquitaine Region, CTTC, 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 fruitful 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.

Key words: Stereolithography, Polymer Derived Ceramic, Silicon oxycarbide, Additive manufacturing

Magic of 3D printed ceramic composites: Structural to Functional

Hui Mei*

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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 Cu_xO 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.

Keywords: 3D printing, Ceramic, Mechanical properties, electromagnetic properties, Catalytic properties, energy storage

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Posters

(Order by topic and by name of main author)

Some abstracts are missing. They will be added after the conference.

Topic 1 – Modeling

Empirically Derived Model for Alloyed Silicon Carbide Microstructural Evolution

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ABSTRACT:

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.

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

Omri Yannay[1,2], Ido Koresh[2], Pini Shekhter[2], Royi Padan[2], Jacob Aboudi[1], and Rami Haj-Ali[1]

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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 2 – Fibers and preforms

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

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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 3 – Interphases

Strengthening and toughening of dense ceramic matrix composites

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Abstract: 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.

Keywords: Ceramic matrix composites; Strengthening and toughening; MAX phases; Interphase; Thermal residual stress

Topic 4 – Oxide/Oxide CMC S

Formulation of oxide suspensions for liquid processing of ceramic matrix composites

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Keywords:

Alumina / Silica / Dispersion / Sedimentation / Rheology

Abstract:

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, $d_{50} = 0.3$ 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 $2.5 \cdot 10^{-6}$ 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 $3Al_2O_3$-$2SiO_2$, 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.

Solutions for CMC

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Gladys BONNE

Abstract

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. The poster will review the properties of the oxide powders suitable for the matrix in Oxide/Oxide CMCs.

Baikowski in CMC field

With its broad range of nanoparticles, Baikowski is a leading supplier of matrix precursors for Oxide/Oxide CMCs.

Precursors solutions developed by Baikowski

To improve the affinity between the fibers and the matrix precursors in CMCs Baikowski is developing several types of oxides: alumina, YAG, mullite and zirconia. The matrix composition must be chosen regarding the properties needed in the final composite: thermal shock resistance, fracture toughness, low thermal conductivity, dielectric transparency … The phasic and chemical purity of the oxides has to be accurately controlled.

To ensure a high sintering reactivity and to control the porosity the oxides particle size distribution must be tight and monomodal and much smaller than the diameter of the ceramic fibers. Baikowski is producing fines high-end oxides (d50 < 0.2 µm).

The solutions developed by Baikowski could address different processes such as slurry for Prepeg or powders easily dispersible. Researches are made to improve the solid loading of the slurries while conserving low viscosity.

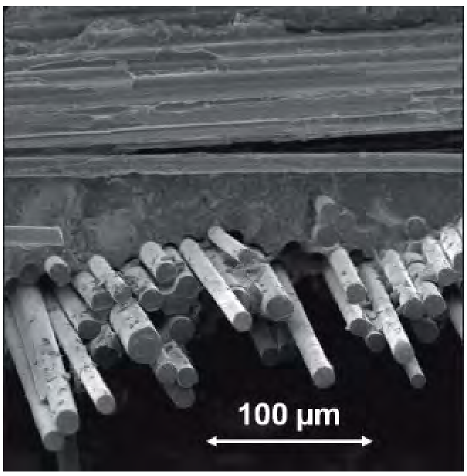

Figure 1 SEM image - oxide/oxide CMC made with Baikowski alumina

Topic 5 – Non-oxide CMC S

High temperature dielectric characterization of SiC-based Ceramic Matrix Composites

Andrea Cintio[1], Roberto D'Ambrosio[2], Andrea Lazzeri[2], Giuseppe Annino[1]

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In microwave-assisted chemical processes, a key quantity is given by the dielectric permittivity $\varepsilon=\varepsilon'-i\varepsilon''$ of the materials to process, since it characterizes the energy absorption from the electromagnetic field. The knowledge of ε 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 (SiC_f/SiC), for various temperatures and infiltration levels. In the investigated SiC_f/SiC, a strong (superlinear) increase of ε' with the density is observed. Moreover, ε' increases monotonously with temperature. A similar behavior is observed with ε'', at least for relatively low densities.

Following, results concerning granular α and β SiC samples with grains of different average size are shown. In this case the measurement method provides an average value of dielectric permittivity $\langle\varepsilon\rangle$ that takes into account the interstitial regions between grains. The α-SiC samples show a relatively constant $\langle\varepsilon'(T)\rangle$ and a relatively low $\langle\varepsilon''(T)\rangle$, decreasing with T. The grain size does not significantly affect the value of $\langle\varepsilon\rangle$, with the exception of an increase of the dielectric losses with T. On the contrary, the β –SiC sample exhibits comparatively higher values of ε' and ε'', both of which increasing with T.

The obtained results show that SiC-based ceramic materials display very different behaviors of ε(T), which depend on the density, the shape, and the crystalline structure. The observed variability in ε(T) confirms the importance of a dielectric characterization at the T of interest.

Keywords: SiC materials, Dielectric characterization, High temperature

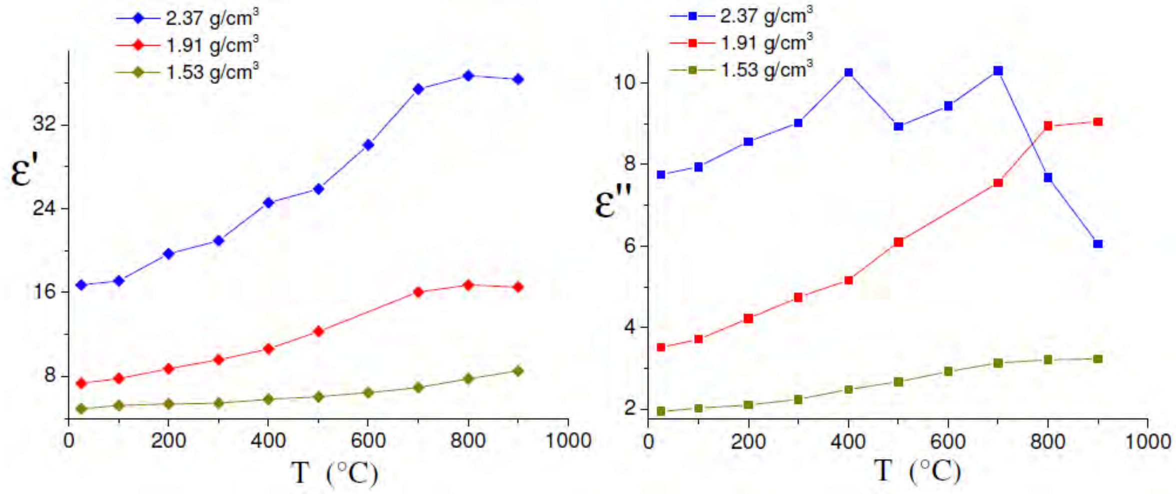

Figure 1 – Real part (left) and imaginary part (right) of the dielectric permittivity of the investigated SiC_f/SiC samples

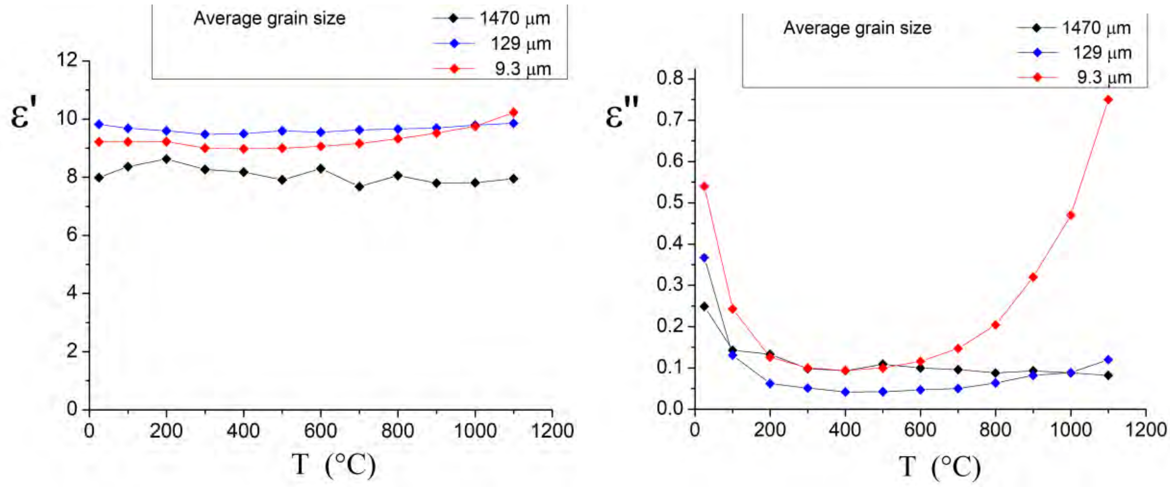

Figure 2 - Real part (left) and imaginary part (right) of the dielectric permittivity of the α-SiC samples for different average grain sizes

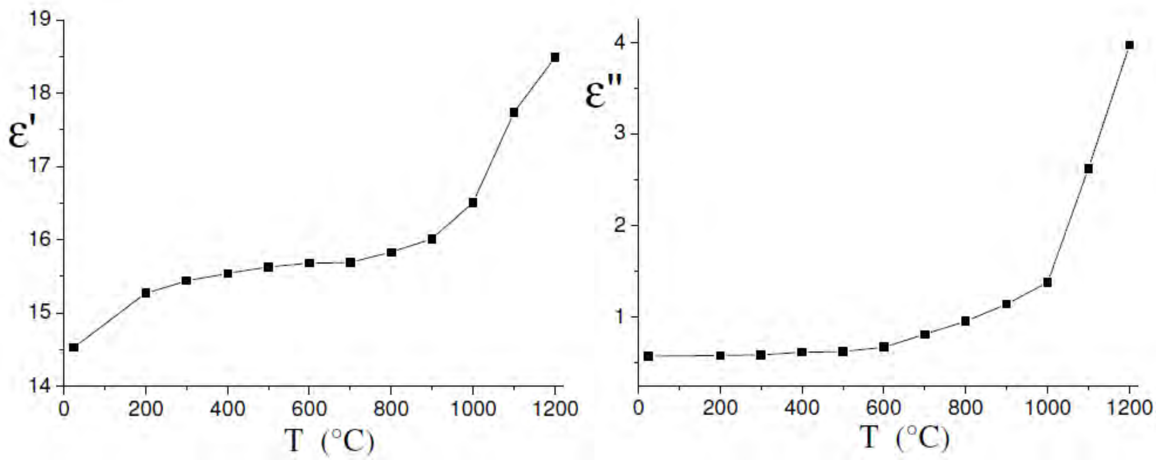

Figure 3 - Real part (left) and imaginary part (right) of the dielectric permittivity of the β-SiC samples synthesized from activated carbon (AC) and silicon (Si)

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

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GEDEON : RP19-0049

Abstract :

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.

LIGHTWEIGHT CERAMIC MATRIX COMPOSITES OF B4C INFILTRATED BY AL-ALLOY

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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 (B_4C, Si_3N_4, 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 (B_4C-Al) prepared by *pressureless infiltration*.

B_4C 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 B_4C 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.

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

Authors

Keywords: C/C-SiC composite, LSI, injection moulding, large-scale production, pyrolysis process

Nier, N.; Ahmad, H.; Gurk, H.; Päßler, E.; Roder, K.; Nestler, D.; Kroll, L.; Wagner, G.

Abstract

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.

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

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Abstract

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 infiltration method and LSI method were found to have improved mechanical properties at high temperatures.

Key words: SiC_f/SiC composite, LSI, SiC slurry, density, high temperature tensile strength

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

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Keywords: Thermoset injection mouldiung; C/C-SiC; mechanical properties;

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 6 – Additive Manufacturing

Additive Manufacturing of Silicon Carbide parts

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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.

Key words: Silicon Carbide, Additive manufacturing, Robocasting, Binder-jetting

Topic 7 – UHTCS & MAXS

Chemical modification of refractory carbides in a gas atmosphere of SiO

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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, Ta_5Si_3 and $TaSi_2$, 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).

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

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Abstract

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.

Key words: Ceramic matrix composites; sand-wind erosion; Oxidation; Ablation property

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

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Abstract

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.

Keywords: Carbides; Monolithic; Fibrous; composites; Fracture mechanisms

SHS/RMI process for the synthesis of Ti_3SiC_2/SiC ceramic matrix composites from macrosized non-powder forms of titanium metal

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A novel approach for the synthesis of Ti_3SiC_2/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 Ti_3SiC_2-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 Ti_3SiC_2/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 Ti_3SiC_2 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).

Recent developments in ZrB_2-based composites research in Iran

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Abstract

As a member of ultrahigh temperature ceramics (UHTCs), zirconium diboride (ZrB_2) 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 ZrB_2-based UHTCs. By searching for the word "ZrB_2" 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 ZrB_2-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.

Keywords: Zirconium diboride; Research; Densification; Additives; Iran.

Topic 8 – EBC/TBC

Thermodynamic calculation of CVD Yttrium Silicate from Y_2O_3-CH_3SiCl_3-CO_2-H_2-Ar System

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Keywords: yttrium silicate; thermodynamics; chemical vapor deposition

SiC_f/SiC composites have many excellent properties, such as low density, high strength, high modulus 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. In this paper, we propose the idea of preparing yttrium silicate-modified SiC/SiC composite matrix by chemical vapor deposition from Y_2O_3-CH_3SiCl_3-CO_2-H_2-Ar system at a lower temperature for a long time. The effect of deposition temperature, pressure, reactant ratio and other process conditions were investigated. The results indicated that the most important factor is the proportion of CO_2 in the gas source. As the proportion of CO_2 increases, the amount of yttrium silicate increases. When $n(CO_2)/n(Y_2O_3+CH_3SiCl_3) \geq 3$, the yttrium silicate product yield tends to be stable. Under the condition of ensuring sufficient CO_2, the H_2 dilution ratio and the total system pressure have little effect on the amount of yttrium silicate, but in order to reduce the by-product C 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. When the temperature is between 1100°C and 1400°C, the yttrium silicate crystal form is stable. And the amount of CO_2 is sufficient, the temperature is higher than 1000°C, the formation of C and SiC phases can be reduced. Obviously, the value of $n(Y_2O_3)/n(Y_2O_3+CH_3SiCl_3)$ has a great influence on the amount of yttrium silicate. When the temperature is higher than 1000°C, the yield of yttrium silicate increases with the increase of $n(Y_2O_3)/n(Y_2O_3+CH_3SiCl_3)$. However, after the value of $n(Y_2O_3)/n(Y_2O_3+CH_3SiCl_3)$ is increased to 0.4, due to the limitation of the software database, we cannot determine the crystal form of yttrium silicate at this time, and it is impossible to determine the change of its amount. This requires experimental judgment. Subsequently, according to the thermodynamic calculation results, X1-Y_2SiO_5 and β-$Y_2Si_2O_7$ were successfully deposited at a lower deposition temperature of 1100°C, a system pressure of 2000Pa, a flow of CH_3SiCl_3 of 39ml/min, a H_2 dilution ratio of 9, and a ratio of $n(CO_2)/n(H_2)$ of 3:10.

Processing of dense rare-earth silicates used as environmental barrier coating

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Keywords: Rare earth silicates, densification, environmental barrier coating

Abstract:

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 (T_m > 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 RE_2O_3-SiO_2 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 RE_2O_3-SiO_2-Al_2O_3 will be considered to obtain a liquid phase to improve the densification of the EBC.

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INVESTIGATION AND PROCESS DEVELOPMENT OF ELECTROPLATED NiCo-Al2O3 ALLOY COMPOSITES

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Abstract

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/dm^2, although lower solid loadings do not alter the results significantly.

Keywords: Bath electroplating, nano-particle, solid loading

Development of hafnia-based materials as potential environmental and thermal barrier for thruster application

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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[1,2].

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 HfO_2 seems to be one of the most promising candidates. The cubic phase stability, obtained through doping with rare earth oxide[3], 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 (Dy_2O_3, Er_2O_3, Gd_2O_3, Lu_2O_3, Y_2O_3 and Yb_2O_3), 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 Y_2O_3 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.

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Topic 9 – PDCS

Dielectric property and interfacial polarization of polymer-derived amorphous silicon carbonitride

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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 10 – Carbon-Carbon Composites

Topic 11 – Thermomechanical behavior and performance

Thermomechanical characterization of 3D needdle Carbon/Carbon composites at very high temperature for space application.

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Ceramic Matrix **C**omposites (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 ([1,2,3,4,5,6]). 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 (µCT) have been carried-out on specimens from 25°C up to 2000°C with a specific device. Coupling X-ray µCT and mechanical tests is a powerful technique [1]. 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 [2, 6].

Results of this *in-situ* tensile tests campaign and first confrontations between experiments and modelling will be presented.

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Topic 12 – NDT and Health Monitoring

Modeling nonlinear shear behaviors of 2D C/SiC z-pinned joint

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Abstract

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.

Keywords

Failure mechanism; Ceramic matrix composite; shear; joint

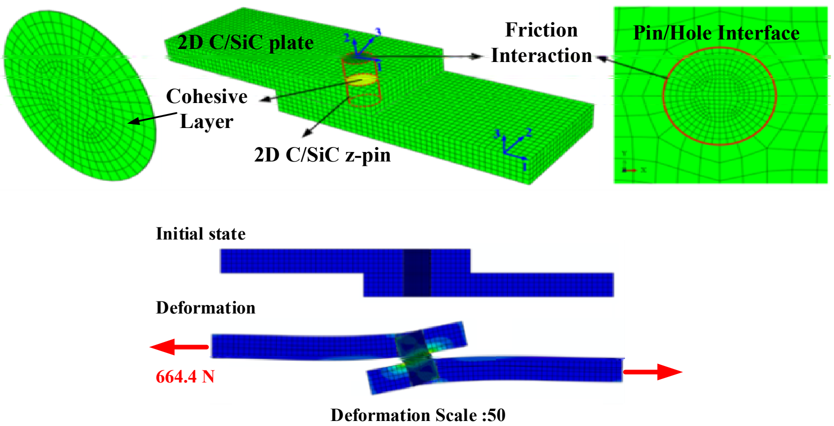

Fig. 1 (a) Finite element model of z-pinned joint, (b) Calculated secondary bending effects.

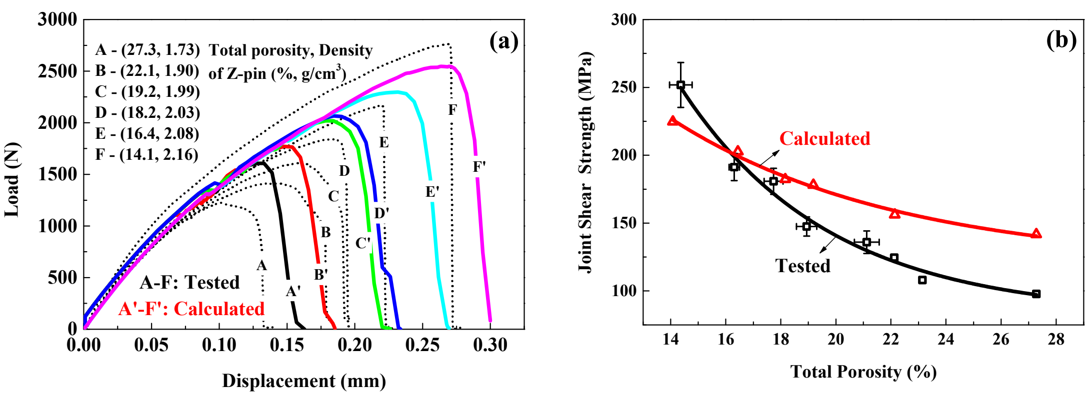

Fig. 2 (a)Tested and calculated tensile load-displacement curves, (b) Comparison between tested and calculated joint shear strengths.

Modeling shear failure mechanisms of 2D C/SiC composite under off-axis loading

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Abstract

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.

Keywords

Failure mechanism; Ceramic matrix composite; Crack; Off-axis loading

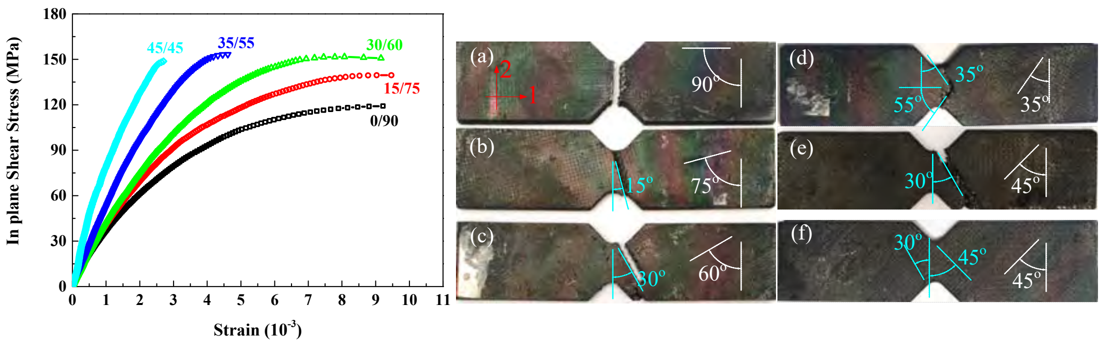

Fig. 1 (a) In-plane shear behaviors of 2D C/SiC composite under off-axis shear loading. (b) Failed Iosipescu samples.

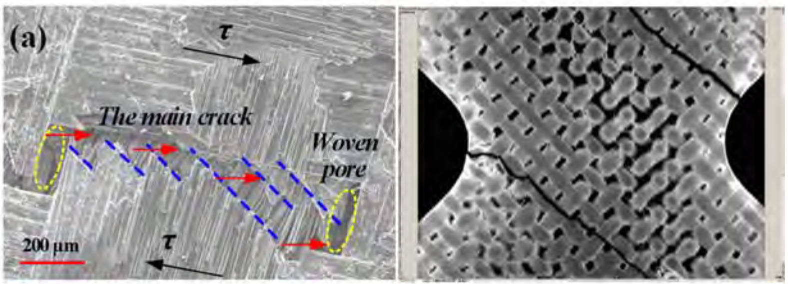

Fig. 2 (a) Periodical matrix cracking of 2D C/SiC composite under 0° shear loading. (b) Crack deflection of 2D C/SiC composite under 45° shear loading.

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Topic 13 – Joining

“RM-Wrap” joining technology for CMC

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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 ($MoSi_2$, $NbSi_2$, $TaSi_2$, $ZrSi_2$ …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 .

Joining and mechanical testing of oxide/oxide ceramic composites

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Efficient joining materials and techniques are of critical importance for the integration of CMCs in high performance structures. Continuous Nextel™ 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.

INTERFACIAL REACTION KINETICS AND MICROSTRUCTURAL EVOLUTION OF C/SiC COMPOSITES TO TITANIUM ALLOY JOINTS

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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.

Keywords: C/SiC composite, Titanium alloy, Vacuum Brazing, Microstructure, Interfacial

Topic 14 – CMC in Space Applications

Topic 15 – Terrestrial Transport & Industrial Applications

Topic 16 – Aeronautic Applications

Topic 17 – Energy Applications

Thermal conductivity estimation of fully ceramic microencapsulated pellets with ZrO_2 as simulated particles

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Abstract

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 β-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.

Keywords: Fully ceramic microencapsulated, thermal conductivity, ZrO2-SiC pellets

1. Introduction

FCM fuel consist of tri-structural isotropic (TRISO) particles embedded in dense silicon carbide (SiC) matrix. TRISO fuel particle technology has been developed and optimized for high temperature gas-cooled reactor (HTGR) over the past five decades[1], while in conventional HTGR fuel these TRISO particles are embedded in graphite matrix. The replacement of matrix material based on the consideration of tremendous merits of SiC.

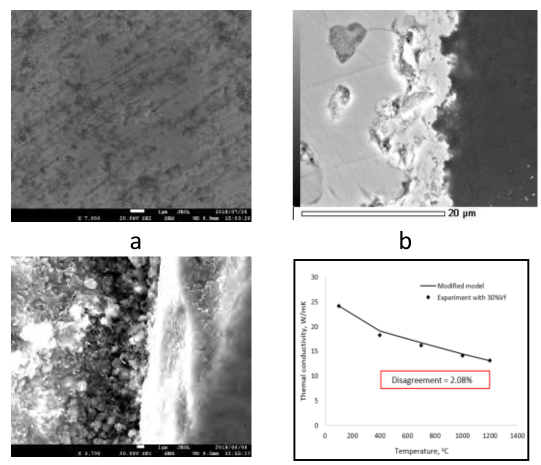
Most of previous studies[2,3] on estimation of FCM thermal conductivity focused on the particle volume fraction inside the matrix. Present work deals with the estimation of thermal conductivity with considering the interaction between components forming fuel pellet. In this work, ZrO_2 (5%Y_2O_3) particles were chosen as simulated of TRISO particles.

2. Experimental

The ZrO_2-SiC FCM fuels with ZrO_2 volume fraction of 0% to 30% and with ZrO_2 particle size of 400 μm were prepared by spark plasma sintering (SPS) method at 2073 K under uniaxial pressure of 92 MPa for 15 min. The SPS pellet then was characterized by laser flash analysis (LFA) to obtain thermal conductivity data, and by scanning electron microscopy with energy dispersive spectroscopy (SEM-EDS) to observe the pellet cross-sectional microstructure.

3. Result and discussion

The existences of pores in the SiC matrix, interfacial layer and gaps between ZrO_2 particles and SiC matrix observed by SEM-EDS (Figure 1a, 1b, 1c) were suspected as the cause of decreasing the pellet thermal conductivity. The formulas were obtained to correct thermal conductivity values for the matrix porosity and particle matrix interface and gaps. These corrections then inserted to the basic equation of Maxwell-Eucken to obtain effective thermal conductivity and plotted with the measured value as shown in Figure 1d in the case of volume fraction of ZrO_2 of 30%.



a b c d

Figure 1. a. SiC matrix pores; b. Interfacial layer; c. gaps between ZrO_2 particles and SiC matrix; d. estimated and measured thermal conductivity ZrO_2-SiC pellet.

4. Conclusion

The simulated FCM pellets were fabricated by SPS in order to obtain the densified samples. The estimation model considering matrix porosity, interfacial layer, and gas conductivity inserted to the basic equation resulting in close agreement between the measured and estimated thermal conductivity.

References

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HT-CMC/10th
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OVERALL CONFERENCE SCHEDULE

8:00 · 9:00 · 10:00 · 11:00 · 12:00 · 13:00 · 14:00 · 15:00 · 16:00 · 17:00 · 18:00 · 19:00

Sunday, Sept. 22

- Registration
- Welcome reception

Monday, Sept. 23

- Registration
- Opening
- Plenary
- Coffee break
- 5 | 13 | 12 | 11 | 3
- Lunch
- 5 | 13 | 12 | 11 | 3
- Coffee break
- 5 | 10 | 12 | 11 | 7

Tuesday, Sept. 24

- Registration
- Plenary
- 5 | 10 | 7 | 11 | 14
- Coffee break
- 5 | 9 | 7 | 11 | 14
- Lunch
- 5 | 9 | 7 | 2 | 14
- Coffee break
- 8 | 9 | 7 | 2 | 16

Wednesday, Sept. 25

- Registration
- Plenary
- 8 | 9 | 1 | 2 | 16
- Coffee break
- 8 | 9 | 1 | 4 | 16
- Lunch
- Poster session
- Bus to Bordeaux
- Bordeaux tour
- Gala dinner

Thursday, Sept. 26

- Registration
- Plenary
- 8 | 17 | 1 | 4 | 15
- Coffee break
- 8 | 17 | 1 | 4 | 15
- Lunch
- 8 | 17 | 1 | 4 | 6
- Coffee break
- 8 | | 1 | 4 | 6
- Concluding remarks

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