

# LINCET 1

Leverhulme International Network on Composites for Extreme Temperatures  
30<sup>th</sup>- 31<sup>st</sup> March, 2017



The Gordon Laboratory



The Leverhulme Trust

## LINCET 1

This Symposium will take place in Downing College, Cambridge (right), on 30<sup>th</sup>/31<sup>st</sup> March, 2017. It will be funded by the Leverhulme Trust, as part of the activities associated with an International Network grant, held in Cambridge University and supported by the Gordon Laboratory in the Materials Science Department there. The Symposium chairs will be Bill Clyne (Cambridge), Robert Vassen (FZ-Jülich) and Dietmar Koch (DLR Stuttgart).



## Symposium Coverage

The network is focused on Composites and Coatings for use in highly demanding environments - particularly at very high temperatures. The programme will be divided into 4 sessions, covering challenges faced in further development of: (a) coating systems, (b) innovative testing techniques, (c) novel types of composite and (d) industrial usage of high temperature composites.

## Format of the Meeting

Attendance is by invitation only and there is no registration fee. Just over 20 delegates will be attending (all prominent in the research fields concerned), including several from industrial firms. There will be a Symposium dinner in the College on the evening of Thursday 30<sup>th</sup> March, followed by talks throughout the following day. There will be 21 talks (all of 20 minutes duration). Lunches and refreshments will also be in this location. Delegates will be accommodated in nearby rooms in the College (right).



## Programme

Thursday 30<sup>th</sup> March

19.00-19.30	Reception in West Lodge
19.30-22.00	Symposium Dinner in West Lodge

Friday 31<sup>st</sup> March

Time	No.	Presenter	Affiliation	Title
<b>Breakfast (Hall) &amp; Registration (West Lodge)</b>				
Session 1: <b>Coatings for High Temperature Use</b> (Chair: <b>Dietmar Koch</b> )				
08.40-09.00	1	Emine Bakan	FZ Jülich	<i>Thermal Spray Deposition of Environmental Barrier Coatings for Si-Based Ceramic Matrix Composites</i>
09.00-09.20	2	Kirsten Bobzin	RWTH Aachen	<i>Coating Developments for Turbomachinery</i>
09.20-09.40	3	Robert Vassen	FZ Jülich	<i>Thermally Sprayed Thermal Barrier Coatings with Columnar Microstructure</i>
09.40-10.00	4	Mark Rainforth	Sheffield University	<i>Design of Coatings for High Temperature Wear Resistance</i>
10.00-10.20	5	Daniel Mack	FZ Jülich	<i>High Temperature Protective Coatings for Variable Load Conditions</i>
10.20-10.40	6	Tobias Kalfhaus	FZ Jülich	<i>Development of Repair Methods for Single Crystalline CMSX-4 with Different Thermal Spray Techniques</i>
<b>Coffee</b>				
Session 2: <b>Novel Testing and Investigation Techniques</b> (Chair: <b>Robert Vassen</b> )				
11.00-11.20	7	Philip Withers	Manchester University	<i>3D and time lapse 3D imaging of high temperature materials degradation</i>
11.20-11.40	8	James Gibson	RWTH Aachen	<i>Towards Operational Temperatures: Nanoindentation of CMSX-4 and an Amdry-386 Bondcoat to 1000C</i>
11.40-12.00	9	Jon Douglas	Frazer-Nash Consultancy	<i>Application of Nuclear Assessment Methods for Remaining Useful Lifetime Prediction of Gas Turbine Components</i>
12.00-12.20	10	Yuan Shi	DLR Stuttgart	<i>Characterization of Hardness and Stiffness of Ceramic Matrix Composites through Instrumented Indentation Test</i>
12.20-12.40	11	Sandra Korte-Kerzel	RWTH Aachen	<i>Low Temperature Plasticity in a Brittle High Temperature Material – Insights in the Effect of Microalloying in MoSi<sub>2</sub> from Nano-mechanical Testing</i>
12.40-13.00	12	James Dean	DPC / Cambridge University	<i>Methodology and Software for Extraction of Plasticity Parameters from Instrumented Indentation</i>
<b>Lunch</b>				
Session 3: <b>Novel Types of High Temperature Composite</b> (Chair: <b>Bill Clyne</b> )				
13.40-14.00	13	Dave Armstrong	Oxford University	<i>Micromechanical Behaviour of SiC/SiC Composites for Next Generation Fuel Cladding</i>
14.00-14.20	14	Jesus Gonzalez-Julian	FZ Jülich	<i>MAX phase/SiC fiber composites</i>
14.20-14.40	15	Laura Silvestroni	CNR-ISTEC Faenza	<i>Ceramic Composites for Extreme Environments: effect of W doping</i>
14.40-15.00	16	Bernd Mainzer	DLR Stuttgart	<i>Microstructural and Mechanical Characterization of Damage Tolerant SiC/SiCN Ceramic Matrix Composites Manufactured via PIP Process</i>
15.00-15.20	17	Vito Leisner	DLR Köln	<i>Development of Environmental Barrier Coatings for SiC/SiC Ceramic Matrix Composites Employing PVD Technologies</i>
15.20-15.40	18	Dietmar Koch	DLR Stuttgart	<i>Manufacturing of Damage Tolerant Ceramic Matrix Composites via Reactive Melt Infiltration for Long Term Applications</i>
<b>Tea</b>				
Session 4: <b>Processing and Industrial Usage of High Temperature Composites</b> (Chair: <b>James Dean</b> )				
16.00-16.20	19	Martin Bram	FZ Jülich	<i>Application of Pressure and Field-Assisted Sintering at the Institute IEK-1</i>
16.20-16.40	20	Stefan Lampenscherf	Siemens	<i>Ceramic Matrix Composites for Gas Turbine Applications</i>
16.40-17.00	21	Lee Marston	Fiberstone	<i>High Temperature Stability and Industrial Usage of "Fiberstone" Metal Fibre Reinforced Ceramic Composites</i>

## Delegates

Name <e-mail>	Affiliation	Downing room	Dietary & Guest
Dave Armstrong <david.armstrong@materials.ox.ac.uk>	Oxford University	30 <sup>th</sup> (D)	Helena Kelly (no gluten/diary)
Emine Bakan <e.bakan@fz-juelich.de>	FZ Jülich	30 <sup>th</sup> & 31 <sup>st</sup> (S)	veg
Kirsten Bobzin <Bobzin@iot.rwth-aachen.de>	RWTH Aachen	30 <sup>th</sup> (S)	
Martin Bram <m.bram@fz-juelich.de>	FZ Jülich	30 <sup>th</sup> & 31 <sup>st</sup> (S)	
Bill Clyne <twc10@cam.ac.uk>	Cambridge University		Gail Clyne (gluten-free)
James Dean <jd362@cam.ac.uk>	Cambridge University		Rachel Moxon
Jon Douglas <j.douglas@fnc.co.uk>	Frazer-Nash Consultancy	30 <sup>th</sup> (S)	
James Gibson <gibson@imm.rwth-aachen.de>	RWTH Aachen	30 <sup>th</sup> (S)	
Jesus Gonzalez-Julian <j.gonzalez@fz-juelich.de>	FZ Jülich	30 <sup>th</sup> & 31 <sup>st</sup> (S)	
Tobias Kalfhaus <t.kalfhaus@fz-juelich.de> Dept. visit	FZ Jülich	30 <sup>th</sup> & 31 <sup>st</sup> (D)	Theresa Wenger
Dietmar Koch <Dietmar.Koch@dlr.de>	DLR Stuttgart	30 <sup>th</sup> & 31 <sup>st</sup> (D)	Ingrid Koch (veg)
Sandra Korte-Kerzel <Korte-Kerzel@imm.rwth-aachen.de>	RWTH Aachen	30 <sup>th</sup> (S)	
Stefan Lampenscherf <stefan.lampenscherf@siemens.com>	Siemens	30 <sup>th</sup> , 31 <sup>st</sup> & 1st (D)	Sybille Lampenscherf
Vito Leisner <vito.leisner@dlr.de> Dept. visit	DLR Köln	30 <sup>th</sup> & 31 <sup>st</sup> (S)	
Daniel Mack <d.e.mack@fz-juelich.de>	FZ Jülich	30 <sup>th</sup> & 31 <sup>st</sup> (S)	
Bernd Mainzer <bernd.mainzer@dlr.de>	DLR Stuttgart	30 <sup>th</sup> & 31 <sup>st</sup> (S)	
Lee Marston <Lee.Marston@dynamic-materials.com>	Fibertech	30 <sup>th</sup> & 31 <sup>st</sup> (S)	
Mark Rainforth <m.rainforth@sheffield.ac.uk>	Sheffield University	30 <sup>th</sup> (S)	veg
Laura Silvestroni <laura.silvestroni@istec.cnr.it> Dept. visit	CNR-ISTEC Faenza	30 <sup>th</sup> & 31 <sup>st</sup> (S)	
Yuan Shi <yuan.shi@dlr.de>	DLR Stuttgart	30 <sup>th</sup> & 31 <sup>st</sup> (S)	
Robert Vassen <r.vassen@fz-juelich.de>	FZ Jülich	29 <sup>th</sup> , 30 <sup>th</sup> & 31 <sup>st</sup> (D)	Andrea Carl
Philip Withers <p.j.withers@manchester.ac.uk>	Manchester University	30 <sup>th</sup> (S)	gluten-free

## Seating Plan for Dinner

	Gail Clyne <sup>§</sup>	Bill Clyne	Robert Vassen	Andrea Carl	
Dietmar Koch					Philip Withers <sup>§</sup>
Ingrid Koch*	James Dean		Mark Rainforth*		Laura Sylvestroni
Vito Leisner	Rachel Moxon		Sandra Korte-Kerzel		Stefan Lampenscherf
Kirsten Bobzin	Dave Armstrong		Jon Douglas		Sybill Lampenscherf
James Gibson			Theresa Kalfhaus		Daniel Mack
Bernd Mainzer	Helena Kelly <sup>§†</sup>		Tobias Kalfhaus		Emine Bakan*
Jesus Gonzalez-Julian	Lee Marston		Yuan Shi		Martin Bram

\* - Vegetarian    § - Gluten-free    † - Dairy-free

## Dinner Menu

- Starter: Potted prawn & brown shrimp, lemon, baby caper dressing
- Main: Mini-rack and pressed shoulder of lamb, pea and Mint puree, crushed parsley potatoes, baby carrots
- Dessert: Baileys crème brulee, almond shortbread (v)
- Veg. Starter: Coco bean and tomato broth, chopped herbs, morel mushrooms
- Veg. Main: Shallot tatin, crispy basil tofu, cauliflower couscous, baby vegetables, roast tomato sauce

# Plan of Downing College



**DOWNING COLLEGE**  
VISITOR PLAN  
dow|2016a

## THE HOWARD CONFERENCE CENTRE

- Howard Theatre
- Grace Howard Room
- Howard Building
- Howard Lodge

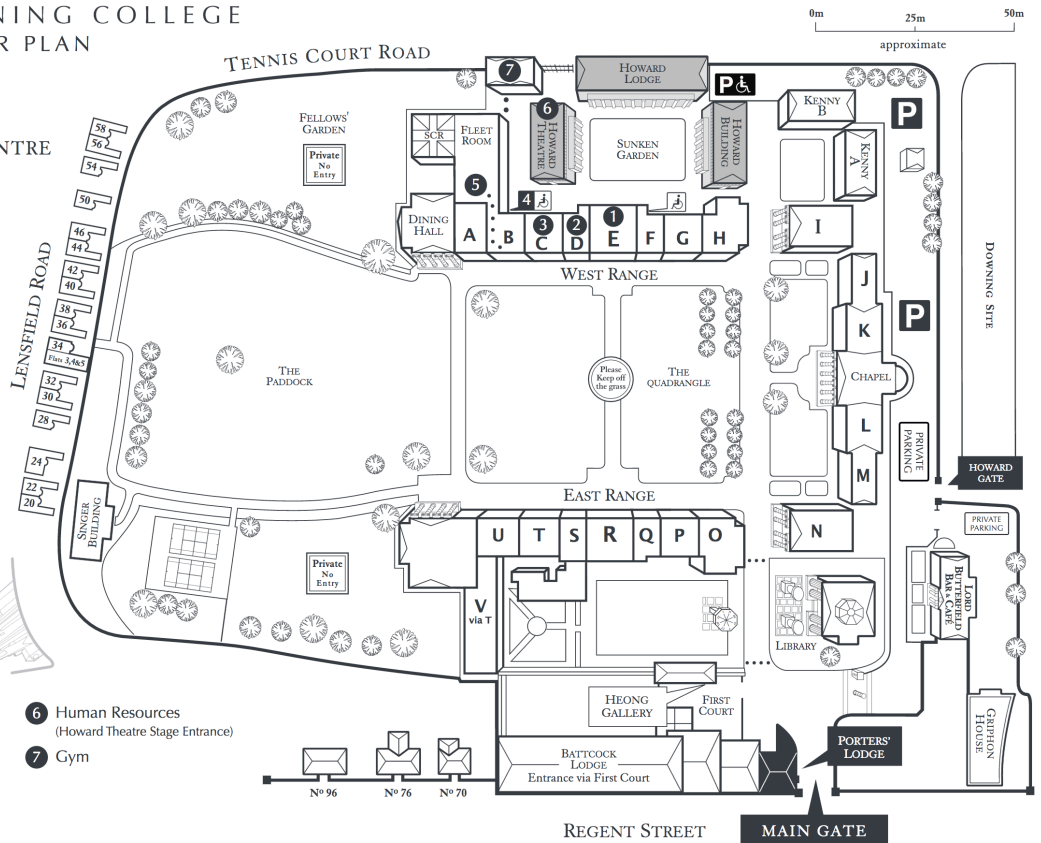
## WEST RANGE (E Staircase)

- West Lodge and Maitland Room
- Music Room and The Tim Cadbury Room

## EAST RANGE (R Staircase)

- Wilkins Room

- 1 Conference Services Office
- 2 College Office
- 3 Development Office
- 4 Level Entrance to A, B & C Staircases
- 5 Bursary  
Catering Managers  
College Accountant  
Tutorial and Admissions
- 6 Human Resources  
(Howard Theatre Stage Entrance)
- 7 Gym



Staircase Letter \_\_\_\_\_

Room Number \_\_\_\_\_

Porters' Lodge 01223 334800

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## Abstracts

### Session 1 - Coatings for High Temperature Use

Talk 1: Friday 31<sup>st</sup> March, 08.40-09.00

#### *Thermal Spray Deposition of Environmental Barrier Coatings for Si-Based Ceramic Matrix Composites*

**E Bakan**

Materials Synthesis and Processing (IEK-1), FZJ, 52425 Jülich, Germany

To enhance the environmental durability of silicon (Si)-based ceramic matrix composites (CMCs) in high pressure, high-gas velocity combustion atmospheres, dense, crack-free, uniform and well-adhered environmental barrier coatings (EBCs) are demanded. This talk will represent an assessment of different thermal spray techniques for deposition of Yb<sub>2</sub>Si<sub>2</sub>O<sub>7</sub> EBCs with desired properties.

Talk 2: Friday 31<sup>st</sup> March, 09.00-09.20

#### *Coating Developments for Turbomachinery*

**K Bobzin, L Zhao, M Öte, T Königstein, S Wiesner & T Liang**

Institute for Surface Engineering, RWTH, Kackertstrasse 15, 52072 Aachen, Germany

In accordance with the key objectives - higher efficiencies and lower emissions - components of modern turbomachinery systems, i.e. stationary and rotating blades, are subject to very high demands, which need to be fulfilled among others with innovative coating solutions. Protective coatings are, therefore, the state of art to ensure functionality of turbomachinery components with prolonged life times. In this presentation, some of research work of Surface Engineering Institute of RWTH Aachen University on the above-mentioned coating solutions is introduced summarily. Firstly, an erosion-resistant coating based on nanocomposite nitride (Ti,Al,Si)N applied by means of high speed physical vapor deposition (HS-PVD) for protection of gas turbine compressor blades and a thick erosion resistant Ni-based composite coating applied by deposition brazing for protection of steam turbine blades will be presented. Subsequently, EB-PVD thermal barrier coatings (TBC) based on different pyrochlore zirconates with multilayer coating architecture will be demonstrated. Furthermore, a highly porous, plasma sprayed TBC, based on Gd<sub>2</sub>O<sub>3</sub>- Yb<sub>2</sub>O<sub>3</sub> co-doped yttria-stabilized zirconia (YSZ), will be introduced. Finally, two oxidation protective coatings for  $\gamma$ -TiAl substrates will be subjected. The first one is an amorphous (Al,Cr)ON coating deposited by means of HS-PVD. The second one is a plasma sprayed coating with a Ti-diffusion barrier interlayer. All of the coatings show promising results with respect to their intended functions..

Talk 3: Friday 31<sup>st</sup> March, 09.20-09.40

#### *Thermally Sprayed Thermal Barrier Coatings with Columnar Microstructure*

**R Vassen**

Materials Synthesis and Processing (IEK-1), FZJ, 52425 Jülich, Germany

This talk will introduce the capabilities and opportunities for imaging the micromechanisms of degradation and failure of high temperature materials. A particular focus will be on time lapse imaging in operando. the talk will include monolithic high temperature materials, coatings, composites and self healing ceramics.

Talk 4: Friday 31<sup>st</sup> March, 09.40-10.00

### *Design of Coatings for High Temperature Wear Resistance*

**WM Rainforth**

Department of Materials Science and Engineering  
Sir Robert Hadfield Building, Mappin Street, Sheffield, S1 3JD, UK

The design of coatings for high temperature wear resistance requires several factors to be considered. It is inevitable that the coating structure will change as a result of the elevated temperature, the contact stresses from the counterface, and the reaction with the environment. The coating must be designed such that the product of these changes also exhibits good friction and wear properties. There are a number of coating architectures and compositions being developed, each optimised for a specific application. For example, TiAlN/VN multilayer coatings exhibit excellent dry sliding wear resistance and low friction coefficient, which is believed to be associated with the formation of self-lubricating Magnéli phases, such as  $V_2O_5$ , although it is now known that the temperature range where this is effective is rather small. In addition, a CrAlYN/CrN coating has been developed which can operate at higher temperatures, which is further enhanced by the use of a nanoscale multilayer CrAlYON/CrON topcoat. An alternative strategy is to apply metal doping to diamond like carbon (DLC) films. For example, the addition of Mo and W to DLC provides a coating with much higher temperature capability through the formation of low friction, wear resistant oxides of Mo and W. This presentation reports detailed analysis of the worn surface structure was undertaken to understand the dynamic changes that result from high temperature frictional contact. The evolution of the wear scar structure and its influence on the dynamic friction coefficient and wear at elevated temperatures is discussed, which points to the future approach to high temperature coating design.

Talk 5: Friday 31<sup>st</sup> March, 10.00-10.20

### *High Temperature Protective Coatings for Variable Load Conditions*

**D Mack**

Materials Synthesis and Processing (IEK-1), FZJ, 52425 Jülich, Germany

Materials in turbines are facing increasingly unsteady conditions under operation. This is due to their diversifying field of application as e.g. in interplay with renewable energy sources. Each set of loading conditions, in terms of e.g. operation temperature or contaminants, present in the combustion atmosphere shows a specific footprint of degradation pathways. Know-how and performance data is available for those specific degradation footprints as to date materials and coatings are developed in respect to each one of those kind of loading scenarios. Less often, materials are assessed at a wider range of conditions where changes of dominant modes of degradation are observed. The demand we are facing today is the design of material systems for volatile load conditions. This requires to predict and to assess the performance under a complex sequence (stochastic mix) of loading phases adding up to the overall degradation. Results are shown on performance and degradation modes of TBC systems under various conditions in cyclic testing. This includes scenarios with sequential vs simultaneous loading with CMAS.

Talk 6: Friday 31<sup>st</sup> March, 10.20-10.40

### *Development of Repair Methods for Single Crystalline CMSX-4 with Different Thermal Spray Techniques*

**T Kalfhaus**

Materials Synthesis and Processing (IEK-1), FZJ, 52425 Jülich, Germany

The durability of modern single-crystal high-temperature turbine blades is limited. The manufacturing of single-crystal turbine blades is a very cost intensive process, therefore the repair of cracked or worn parts is of great interest. The thermal spray techniques VPS (Vacuum Plasma Spray), HVOF (High Velocity Oxygen Fuel) and Cold Spray were tested with CMSX-4 powder on single crystalline CMSX-4 substrate to identify their potential for repair processes.

Session 2 - Novel Testing and Investigation Techniques

Talk 7: Friday 31<sup>st</sup> March, 11.00-11.20

**3D and time lapse 3D imaging of high temperature materials degradation**

**PJ Withers**

Sir Henry Royce Institute for Advanced Materials, The School of Materials  
University of Manchester, Oxford Road, Manchester, M13 9PL, UK

X-ray computed tomography (CT) is capable of providing sub-micron spatial resolution non-destructively. The brightness of synchrotron X-ray sources enables 3D image frame rates as fast as 10Hz, while lab.-based systems enable the time lapse imaging of degradation processes that take place over days or even months. This makes x-ray imaging a very useful tool for study of the behaviour of coatings and composites. In this talk, I will introduce the capabilities of the method and then present a number of case studies showing how it can provide information on the accumulation and healing of damage under conditions representative of *in operando* conditions. Examples will include time-lapse studies of:

- *Thermal cycling of thermal barrier coatings, showing the build-up of the thermally grown oxide and the accumulation of coating damage*
- *Self-healing of cracks in MAX phase ceramics at 1050 °C*
- *Growth of fatigue cracks in Ti/SiC metal matrix composites*
- *Highly ablation resistant  $Zr_{0.8}Ti_{0.2}C_{0.74}B_{0.26}$  carbide for protecting C/C composites under extremely oxidizing environments up to 3000 °C.*

Talk 8: Friday 31<sup>st</sup> March, 11.20-11.40

**Towards Operational Temperatures: Nanoindentation of CMSX-4 and an Amdry-386 Bondcoat to 1000 °C**

**J Gibson**

Institute for Metallurgy & Metal Physics, RWTH, 52056 Aachen, Germany

With nickel-based superalloys reaching their limit in high-temperature gas turbines, further refinement and new alloys are required with improved mechanical properties. Small-scale mechanical testing – particularly nanoindentation – is of great benefit to alloy development, allowing hardness to be measured on small volumes of newly-developed materials.

Talk 9: Friday 31<sup>st</sup> March, 11.40-12.00

**Application of Nuclear Assessment Methods for Remaining Useful Lifetime Prediction of Gas Turbine Components**

**J Douglas**

Frazer-Nash Consultancy, Stonebridge House, Dorking Business Park, Dorking, RH4 1HJ, UK

Exhaustion of available ductility provides a basis for high temperature component lifetime assessments in the civil nuclear industry. The method has been developed over the last fifty years to cope with steel components subject to creep, fatigue and creep-fatigue. Gas turbine components have been developed primarily for creep resistance, due to the nature of the anticipated design cycle for base load operation. However, the recent introduction of renewable energy to the European grid has resulted in the need for flexible, responsive operation of gas power plant. Units designed for three cycles per year now see three cycles per day. This presentation shows how methods developed in the nuclear industry have been adopted to predict remaining useful lives of high integrity superalloy components in gas power plant.



Talk 10: Friday 31<sup>st</sup> March, 12.00-12.20

***Characterization of Hardness and Stiffness of Ceramic Matrix Composites through Instrumented Indentation Test***

**Y Shi**

Institute of Structures and Design, German Aerospace Centre, Pfaffenwaldring 38-40  
70569 Stuttgart, Germany

The determination of the hardness allows useful qualitative conclusions regarding the mechanical properties of materials. It is one of the most economical characterization methods, since this can be carried out with little effort by small plates. However, the characterization of hardness of ceramic matrix composites (CMCs) is a challenge due to their heterogeneous structure and high level of hardness and has thus rarely been investigated. In this work, the experimental procedure and results of determination of the hardness of CMCs using diamond sphere are presented. Several CMCs with different fibers and with porous and dense matrices were investigated. The instrumented indentation testing method was applied with load transducer and displacement sensor. The visual load-unload curve generated during the indentation process was analyzed to determine the hardness and stiffness of the samples. The test load and the number of load-unload-cycles were varied for selected materials in order to provide recommendations for a suitable experimental procedure, which should be applicable to other CMCs.

Talk 11: Friday 31<sup>st</sup> March, 12.20-12.40

***Low Temperature Plasticity in a Brittle High Temperature Material – Insights in the Effect of Microalloying in MoSi<sub>2</sub> from Nano-mechanical Testing***

**S Korte-Kerzel**

Institute for Metallurgy & Metal Physics, RWTH, 52056 Aachen, Germany

One of the major drawbacks of candidate materials for high temperature application is often their brittleness at low temperatures and molybdenum di-silicide is no exception to this. Previous research on micro-alloying has shown that alloying can lead to appreciable plasticity even of single crystals along their most brittle [001] direction. Here, a first attempt will be shown to explicitly characterize how this occurs. Using Ta as a previously little studied alloying element, it is shown how micro-compression and indentation in correlation with EBSD and TEM can shed light on the plasticity of even the most brittle crystals. In this way, a first estimate of the yield stress for deformation in [001] direction at room temperature can be obtained and compared with that in the pure and alloyed material. It is shown that the increase in ductility observed previously is in fact due to activation of an additional slip system ( $\{110\}\langle 111\rangle$ ), not the reduction of the CRSS on  $\{013\}\langle 331\rangle$ .

Talk 12: Friday 31<sup>st</sup> March, 12.40-13.00

*Methodology and Software for Extraction of Plasticity and Creep Parameters from Instrumented Indentation Data*

**J Dean & TW Clyne**

Dept. of Materials Sci., Univ. of Cambridge, 27 Charles Babbage Road, Cambridge CB3 0FS, UK  
& Double Precision Consultancy Ltd, Cambridge, UK

Indentation is a simple and attractive procedure and, provided the tested volume is large enough for its response to be representative of the "bulk", it can in principle be used to obtain material properties. Young's modulus and hardness values are readily obtained from single indentation experimental data, using analytical expressions. However, elastic properties are of limited interest and hardness is not a "genuine" property - it does depend on plasticity parameters (yield stress and work-hardening characteristics), but they cannot be obtained from a hardness value. Procedures are in use that allow extraction of creep parameters from indentation (displacement-time) data, via use of analytical expressions, but in fact they are flawed and unreliable. The only methodology offering real promise of reliability is inverse FEM modelling - ie iterative FEM simulation of indentation, using trial, and then revised, values of the relevant property parameters, until optimal agreement is reached between modelled and experimental outcomes (eg load-displacement or displacement-time plots). This is conceptually simple, but challenging in detail, since robust algorithms are needed to characterize the "goodness of fit", converge on best-fit parameter combinations and establish the reliability and uniqueness of the solutions obtained. Recent progress in this area will be summarized and it will be shown that, by screening on different sections of a single load-displacement plot from a spherical indenter, plasticity parameter values can be reliably obtained. User-friendly software packages (available on-line) are now available for implementation of this procedure. The prospects for extending this methodology to creep parameters will also be briefly discussed.

*Session 3 - Novel Types of High Temperature Composite*

Talk 13: Friday 31<sup>st</sup> March, 13.40-14.00

***Micromechanical Behaviour of SiC/SiC Composites for Next Generation Fuel Cladding***

**D Armstrong**

Department of Materials, Oxford University, Parks Road, Oxford OX1 3PH, UK

Silicon carbide is a leading candidate material for the use in novel accident-tolerant fuel cladding due to its favorable properties, in particular reduced (compared to Zircaloy) oxidation under accident conditions, as well as good neutronic performance, high temperature strength and stability under irradiation. It is suggested to be used in the form of SiC-fiber reinforced SiC-matrix (SiC-SiC) composite. Highly non-uniform and anisotropic nature of the composite materials means that, in order to reliably model their behavior, knowledge of the individual properties of fiber and matrix, and, crucially, the fiber-matrix interfaces, is required. Micromechanical testing techniques, such as micro-cantilever beam fracture, allow determination of such localized properties. This contribution, for the first time, reports the results of micromechanical measurements, coupled with microstructural characterization, on SiC-SiC composite material. Material used in this study was provided by General Atomics. It consists of the commercially available Tyranno SiC fiber weaved reinforcement structure and matrix grown in-situ using the chemical vapour infiltration (CVI) technique. General structure of the composite, including fiber arrangement and porosity, was assessed using scanning electron microscopy (SEM) and X-ray tomography. Microstructure of fibers and matrix was characterized with electron backscattered diffraction (EBSD), transmission electron microscopy (TEM) and transmission Kikuchi diffraction (TKD) techniques. Micromechanical studies included hardness measurements on fibers and matrix performed with nanoindentation, and interfacial fracture tests using focused ion beam (FIB) manufactured microcantilevers and miniature fiber pushout tests. Finally, the effect of heavy ion irradiation on silicon carbide is studied using TEM and nanoindentation

Talk 14: Friday 31<sup>st</sup> March, 14.00-14.20

***MAX Phase/SiC Fiber Composites***

**J Gonzalez-Julian**

Materials Synthesis and Processing (IEK-1), FZJ, 52425 Jülich, Germany

MAX phases are excellent candidates for high temperature application (> 1000°C), due to their unique combination of properties, bridging the gap between ceramics and metals. As ceramics, they are lightweight, and show excellent oxidation and corrosion resistances; meanwhile as metals, MAX phases present good damage tolerance, and high thermal and electrical conductivities. Incorporation of fibers to improve properties – mainly the mechanical ones – has been extensively explored in advanced ceramic systems, leading to ceramic matrix composites (CMCs). However, investigations of CMCs based on MAX phases are very limited, despite their large potential for high temperature applications. In this work, processing of the MAX composites containing short SiC fibers and their mechanical and tribological response are presented. In addition, preliminary results about the oxidation response at high temperature of the monolithic and composite materials will be shown.

Talk 15: Friday 31<sup>st</sup> March, 14.20-14.40

***Ceramic Composites for Extreme Environments: Effect of W Doping***

**L Silvestroni**

CNR-ISTEC, Institute of Sci. & Technol. for Ceramics, Via Granarolo 64, I-48018 Faenza, Italy

Among materials able to withstand ablation in extreme environments, the class of compounds commonly known as *Ultra-High Temperature Ceramics (UHTCs)* is a potential candidate, thanks to melting temperatures above 3000°C. However, research on UHTCs has generally stumbled across two major issues: the low fracture toughness and the oxidation resistance. Recent advancements in high temperature mechanical properties have been achieved through addition of W-based compounds, thanks to the formation of highly refractory secondary phases and to a particular configuration of the matrix grains. On the other hand, in order to increase the intrinsic brittleness, long and short fibers have been added to UHTC matrices. However, cautious choice of the sintering parameters must be paid, owing to the tendency of the fibers to react with the matrix at increasing temperatures, change their characteristic structure and thus lose their properties. This work presents a series of UHTCs exhibiting outstanding high temperature mechanical strength, or improved fracture toughness and oxidation resistance. In particular, the microstructure evolution upon sintering or oxidation has been studied by SEM and TEM and correlated to the specific thermo-mechanical performances.

Talk 16: Friday 31<sup>st</sup> March, 14.40-15.00

***Microstructural and Mechanical Characterization of Damage Tolerant SiC/SiCN Ceramic Matrix Composites Manufactured via PIP Process***

**B Mainzer**

Institute of Structures and Design, German Aerospace Centre, Pfaffenwaldring 38-40  
70569 Stuttgart, Germany

Silicon carbide fiber reinforced SiC composites offer performance advantages such as higher temperature capability and significant lower density over the currently used metallic superalloys. In this work silicon carbide fiber reinforced nitrogenous SiC composites (SiC/SiCN) were manufactured by polymer infiltration and pyrolysis. As precursor a polyvinylsilazane with low viscosity was infiltrated via resin transfer molding into Tyranno SA3 fiber preforms, cured and pyrolyzed. Due to process induced shrinkage several re-infiltration and pyrolysis steps had to be performed until an acceptable porosity below 5% was achieved. The matrix precursor was investigated in terms of thermal behavior via differential scanning calorimetry and rheology measurement to find the ideal temperatures for the polymer infiltration step. Pure matrix specimens were cured and pyrolyzed. The densification during pyrolysis was investigated in terms of thermogravimetric analysis, He gas pycnometry and X-ray diffraction. Finally the microstructure of the composite and the quality of the infiltration process was characterized by SEM and  $\mu$ CT. To determine the mechanical properties of the SiC/SiCN composites, samples were tested by means of 3-point bending.

Talk 17: Friday 31<sup>st</sup> March, 15.00-15.20

***Development of Environmental Barrier Coatings for SiC/SiC Ceramic Matrix Composites Employing PVD Technologies***

**V Leisner**

DLR Köln, Germany

Silicon carbide ceramic matrix composites (SiC/SiC-CMCs) are promising materials for application in the hot section of gas turbines. SiC forms a protective SiO<sub>2</sub> scale (TGO), which sufficiently protects the underlying material from oxidation at high temperatures in dry air. However, if rapidly flowing water vapour in a combustion environment is present, the TGO is subjected to severe volatilization by formation of silicon hydroxide. Thus, an environmental barrier coating (EBC) system is required with thermochemically compatible interfaces and a coefficient of thermal expansion (CTE) matching the SiC/SiC-CMC. In this study, coatings were fabricated by magnetron sputtering and reactive magnetron sputtering, respectively, with silicon-based bond coats, Y<sub>2</sub>Si<sub>2</sub>O<sub>7</sub>, and Y<sub>2</sub>SiO<sub>5</sub> or combinations thereof. The coating architecture was designed namely to minimize chemical interactions among different layers and with a strain tolerant microstructure. Emphasis was put on both interfacial chemical reactions and phase evolution.

Talk 18: Friday 31<sup>st</sup> March, 15.20-15.40

*Manufacturing of Damage Tolerant Ceramic Matrix Composites via Reactive Melt Infiltration for Long Term Applications*

**D Koch**

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70569 Stuttgart, Germany

The reactive melt infiltration process is a versatile route to manufacture non-oxide fiber reinforced ceramics (CMC). In general, a porous preform is manufactured reinforced with either Carbon or SiC fibers and a matrix with a specific pore morphology for the succeeding infiltration process. Depending on the composition of the reactive alloy melt and the desired final matrix state Carbon or other filler particles as borides are introduced in the porous matrix. Ideally, all constituents react in the desired manner with the alloy to a more or less dense matrix. The process strategy to manufacture SiC/SiC as well as ultra high temperature ceramic matrix composites UHTCMC is shown and key aspects are discussed. One issue is that the fiber-matrix interphase has to be designed in order to survive the reactive melt infiltration. Additionally the interphase needs to keep low strength for sufficient crack deflection, in order to prevent brittle failure of the composite.

*Session 4 - Processing and Industrial Usage of High Temperature Composites*

Talk 19: Friday 31<sup>st</sup> March, 16.00-16.20

*Application of Pressure and Field-Assisted Sintering at the Institute IEK-1*

**M Bram**

Materials Synthesis and Processing (IEK-1), FZJ, 52425 Jülich, Germany

The Institute of Energy and Climate Research (IEK-1: Materials Synthesis and Processing) of Forschungszentrum Juelich GmbH is equipped with pressure and field assisted sintering technologies including Hot Isostatic Pressing (HIP), Hot Pressing (HP) and Field Assisted Sintering Technology/Spark Plasma Sintering (FAST/SPS). These technologies are usually applied for the densification of ceramic and metal powders as well as composites thereof. Furthermore, HIP technology can be used to close the residual porosity of net shaped parts, which were made by conventional powder processing or additive manufacturing. In the presentation, the different pressure and field assisted sintering technologies mentioned before are introduced. Additionally, some examples of current institute's research based on these technologies in the field of transparent ceramics, electrochemical devices and ceramic matrix composites are given.

Talk 20: Friday 31<sup>st</sup> March, 16.20-16.40

*Ceramic Matrix Composites for Gas Turbine Applications*

**S Lampenscherf**

Siemens AG, CT MM2, Otto-Hahn-Ring 6, Munich 81730, Germany

The usage of CMCs offers the potential to significantly increase efficiency of future power generation gas turbines. In this talk I will discuss opportunities and challenges related to application of CMC in GT hot gas path components.

Talk 21: Friday 31<sup>st</sup> March, 16.40-17.00

*High Temperature Stability and Industrial Usage of "Fiberstone" Metal Fibre Reinforced Ceramic Composites*

**LW Marston & TW Clyne**

Fiberstone Products Ltd, Brookhill Road, Pinxton, Nottingham, NG16 6NT, UK  
& Dept. of Materials Sci., Univ. of Cambridge, 27 Charles Babbage Road, Cambridge CB3 0FS, UK

Fiberstone is a commercially-available ceramic matrix composite material. It contains relatively coarse metallic fibres dispersed randomly in a matrix that is predominantly alumina. It can be "cast" to shape by infiltration of a ceramic slurry into an assembly of fibres contained within a mould. It has a high toughness, with the work of fracture arising predominantly from pull-out and plastic deformation of fibres bridging the crack plane. A model is presented for prediction of the fracture energy. Good agreement with experiment is observed, and there is scope for the fracture energy levels to be high ( $\sim 20 \text{ kJ m}^{-2}$ ). Higher toughness levels are both predicted and observed for coarser fibres, up to a practical limit for the fibre diameter of the order of 0.5 mm. Other deductions are also made concerning strategies for optimisation of the toughness. Results are also presented concerning the effect of heat treatment on toughness and it is shown that use of highly oxidation-resistant fibres, such as AISI310, allows good toughness to be retained after severe heat treatments (tens of hours at temperatures of up to 1170°C). Work is also described in which HIPing has been employed to reduce the level of porosity in these materials, which is expected to further improve the high temperature stability. Some examples are shown of industrial usage under demanding high temperature conditions.