LINCET 2

Leverhulme International Network on Composites for Extreme Temperatures

4th- 5th April, 2019

The Gordon Laboratory

HELMHOLTZ | ASSOCIATION

The Leverhulme Trust

LINCET 2

This Symposium will take place in Downing College, Cambridge (right), on 4th/5th April, 2019. It will be funded by the Leverhulme Trust, as part of the activities associated with an International Network grant, and also by the Helmholtz Association. The Symposium chairs will be Bill Clyne (Cambridge), James Dean (Cambridge) and Robert Vassen (FZ-Jülich).

Symposium Coverage

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

Format of the Meeting

Attendance is by invitation only and there is no registration fee. A total of 26 delegates will be attending (all prominent in the research fields concerned), including several from industry. There will be a Symposium dinner in the College on the evening of Thursday 4th April, followed by talks throughout the following day. There will be 18 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 4th April

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

Friday 5th April

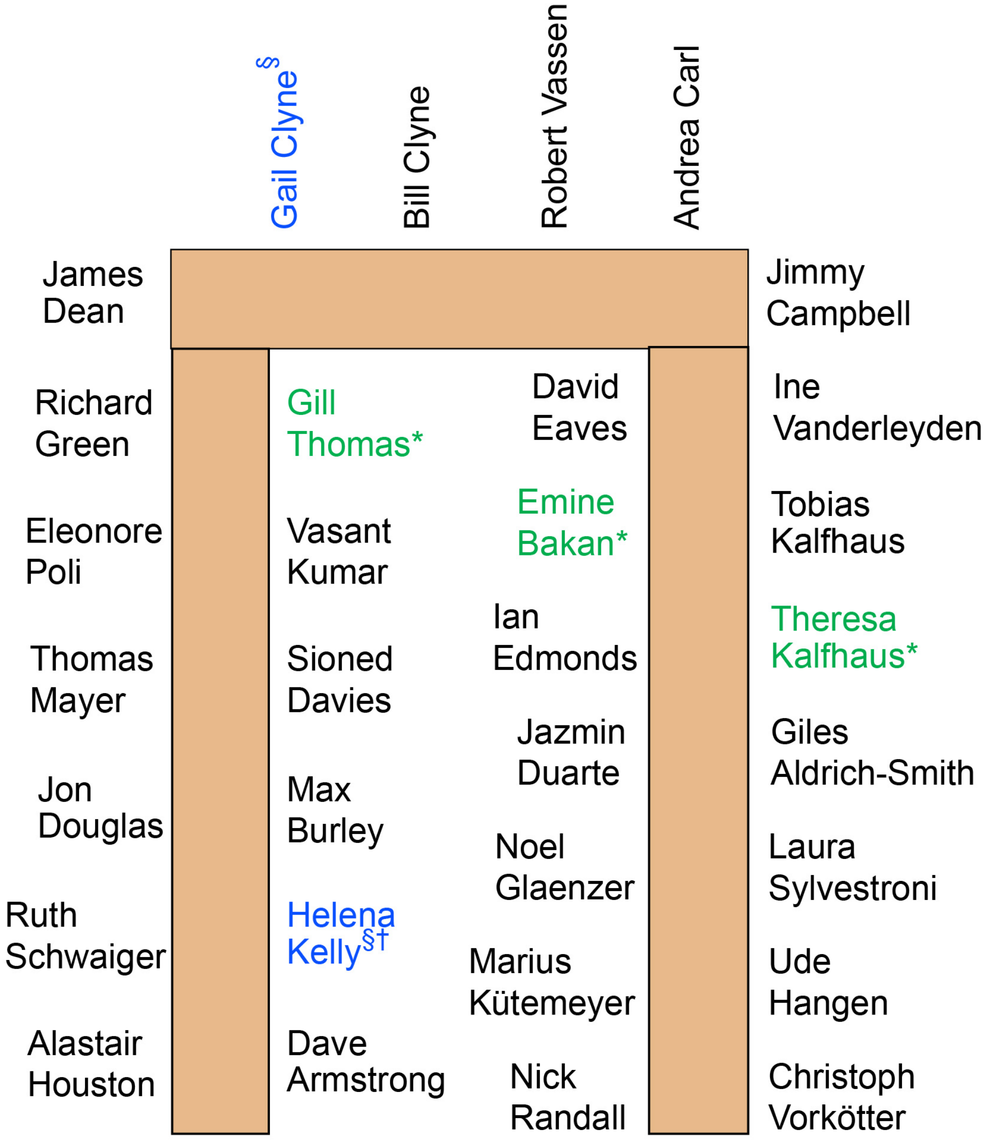
Time	No.	Presenter	Affiliation	Title
08.00-09.00		**Breakfast (Hall) & Registration (Grace Howard Room): Sessions upstairs (Howard Lecture Theatre)**		
09.00-10.40		Session 1: **Materials and Systems in Demanding Industrial Environments** (Chair: **Robert Vassen**)		
09.00-09.20	**1**	David Eaves	Westinghouse	*Developments in Coating Technology at Westinghouse for Nuclear Applications*
09.20-09.40	**2**	Richard Green	Solar Turbines	*Condition Based Engineering for Digital Asset Management*
09.40-10.00	**3**	Jon Douglas	Frazer-Nash Consultancy	*Materials Testing Requirements for Flexible Operation of Gas Turbines*
10.00-10.20	**4**	Thomas Mayer	ZHAW	*Microstructural Evolution in Steam Turbine Components – A Starting Point for More Realistic Lifetime Prediction Methods*
10.20-10.40	**5**	James Dean	Plastometrex / Cambridge University	*Indentation Plastometry for NDT Mapping and In Situ Monitoring*
10.40-11.10		**Coffee (Grace Howard Room)**		
11.10-12.50		Session 2: **Novel Testing and Investigation Techniques** (Chair: **James Dean**)		
11.10-11.30	**6**	Thomas Chudoba	ASMEC	*High Resolution Micro-wear Tests of DLC Coatings in a Temperature Range from RT up to 400°C*
11.30-11.50	**7**	Ude Hangen	Bruker	*Dispersion of mechanical properties at nanoscale studied by indentation mapping*
11.50-12.10	**8**	Ruth Schwaiger	Karlsruhe IT	*Thermally Activated Dislocation Plasticity in BCC Metals and Alloys studied by High-temperature Nanoindentation*
12.10-12.30	**9**	Nick Randall	Alemnis	*Recent Innovation in the Field of In-situ Micro- and Nano-scale Mechanical Testing*
12.30-12.50	**10**	Jimmy Campbell	Cambridge University	*Obtaining Miller-Norton Creep Parameters from Indentation Testing*
12.50-13.40		**Lunch (Grace Howard Room)**		
13.40-13.50		**Group Photo (outside Howard Building)**		
13.50-15.30		Session 3: **Novel Composite Systems for High Temperature (Chair: Jimmy Campbell)**		
13.50-14.10	**11**	Alastair Houston	Cambridge University	*Hybrid Particle-Fibre Ceramic Composite Materials for use as Diesel Particulate Filters*
14.10-14.30	**12**	Laura Silvestroni	CNR-ISTEC Faenza	*C3HARME - Next Generation Ceramic Composites for Combustion Harsh Environment and Space*
14.30-14.50	**13**	Ian Edmonds	Rolls Royce	*Ceramic Matrix Composites for Aerospace – Challenges & Opportunities*
14.50-15.10	**14**	Marius Kütemeyer	DLR Stuttgart	*Challenges during the Fabrication of a Reactive Melt Infiltrated Ultra High Temperature Ceramic Matrix Composite*
15.10-15.30	**15**	Dave Armstrong	Oxford University	*Understanding Interfacial Failure in SiC/SiC Composites through Micro-mechanical Testing*
15.30-16.00		**Tea (Grace Howard Room)**		
16.00-17.00		Session 4: **Surface Protection (Chair: Bill Clyne)**		
16.00-16.20	**16**	Emine Bakan	FZ Jülich	*Environmental Barrier Coatings for SiC/SiC CMCs: Current Progress in Jülich*
16.20-16.40	**17**	Jazmin Duarte	MPI Düsseldorf	*Hydrogen-metal Interactions by In-situ and Ex-situ Methods*
16.40-17.00	**18**	Tobias Kalfhaus	FZ Jülich	*Repair of Ni-Superalloys using Thermal Spray*

Delegates

Name <e-mail>	Affiliation	Downing room etc	Dietary & Guest
Giles Aldrich-Smith <giles.aldrich-smith@awe.co.uk>	AWE	4th only / no parking	-
Dave Armstrong <david.armstrong@materials.ox.ac.uk>	Oxford University	4th only / no parking	Helena Kelly (no gluten/diary)
Emine Bakan <e.bakan@fz-juelich.de>	FZ Jülich	4th & 5th / no parking	veg
Max Burley <jc682@cam.ac.uk>	Cambridge University	-	Sioned Davies
Jimmy Campbell <jc682@cam.ac.uk>	Cambridge University	-	Ine Vanderleyden
Thomas Chudoba <t.chudoba@asmec.de>	ASMEC	4th & 5th / no parking	-
Bill Clyne <twc10@cam.ac.uk>	Cambridge University	-	Gail Clyne (gluten-free)
James Dean <jd362@cam.ac.uk>	Cambridge University	parking	
Jon Douglas <j.douglas@fnc.co.uk>	Frazer-Nash Consultancy	4th & 5th / parking	-
Jazmin Duarte <j.duarte@mpie.de>	MPI Düsseldorf	4th & 5th / no parking	-
David Eaves <eavesdg@westinghouse.com>	Westinghouse	4th only / no parking	-
Ian Edmonds <Ian.edmonds@rolls-royce.com>	Rolls Royce	4th only / parking	-
Noel Glaenzer < ng362@cam.ac.uk>	Solar / Cambridge U.	-	-
Richard Green <Green_Richard_J@solarturbines.com>	Solar Turbines	4th & 5th / no parking	-
Ude Hangen <Ude.Hangen@bruker.com>	Bruker	4th & 5th / no parking	-
Alastair Houston <ajh242@cam.ac.uk>	Cambridge University	-	-
Tobias Kalfhaus <t.kalfhaus@fz-juelich.de>	FZ Jülich	4th only / no parking	Theresa Kalfhaus (veg)
Vasant Kumar <rvk10@cam.ac.uk>	Cambridge University	parking	Gill Thomas (veg)
Marius Kütemeyer <Marius.kuetemeyer@dlr.de>	DLR Stuttgart	4th only / no parking	-
Thomas Mayer <mayt@zhaw.ch>	ZHAW	4th only / no parking	-
Eleonore Poli <ecp40@cam.ac.uk>	Cambridge University	-	-
Nick Randall <nicholas.randall@alemnis.ch>	Alemnis	4th only / no parking	-
Ruth Schwaiger <ruth.schwaiger@kit.edu>	Karlsruhe IT	4th & 5th / no parking	-
Laura Silvestroni <laura.silvestroni@istec.cnr.it>	CNR-ISTEC Faenza	4th only / no parking	-
Robert Vassen <r.vassen@fz-juelich.de>	FZ Jülich	4th only / no parking	Andrea Carl
Christoph Vorkötter <c.vorkoetter@fz-juelich.de>	FZ Jülicht	4th only / no parking	-

Delegates should note that rooms will be available for occupation from 2pm on Thursday 4^{th} April. Rooms should be vacated by 9am on the day of departure. Meeting Room 1 (in the Howard Lecture Theatre Building) will be available for storage of luggage etc throughout the day on Friday 5^{th} April.

Seating Plan for Dinner

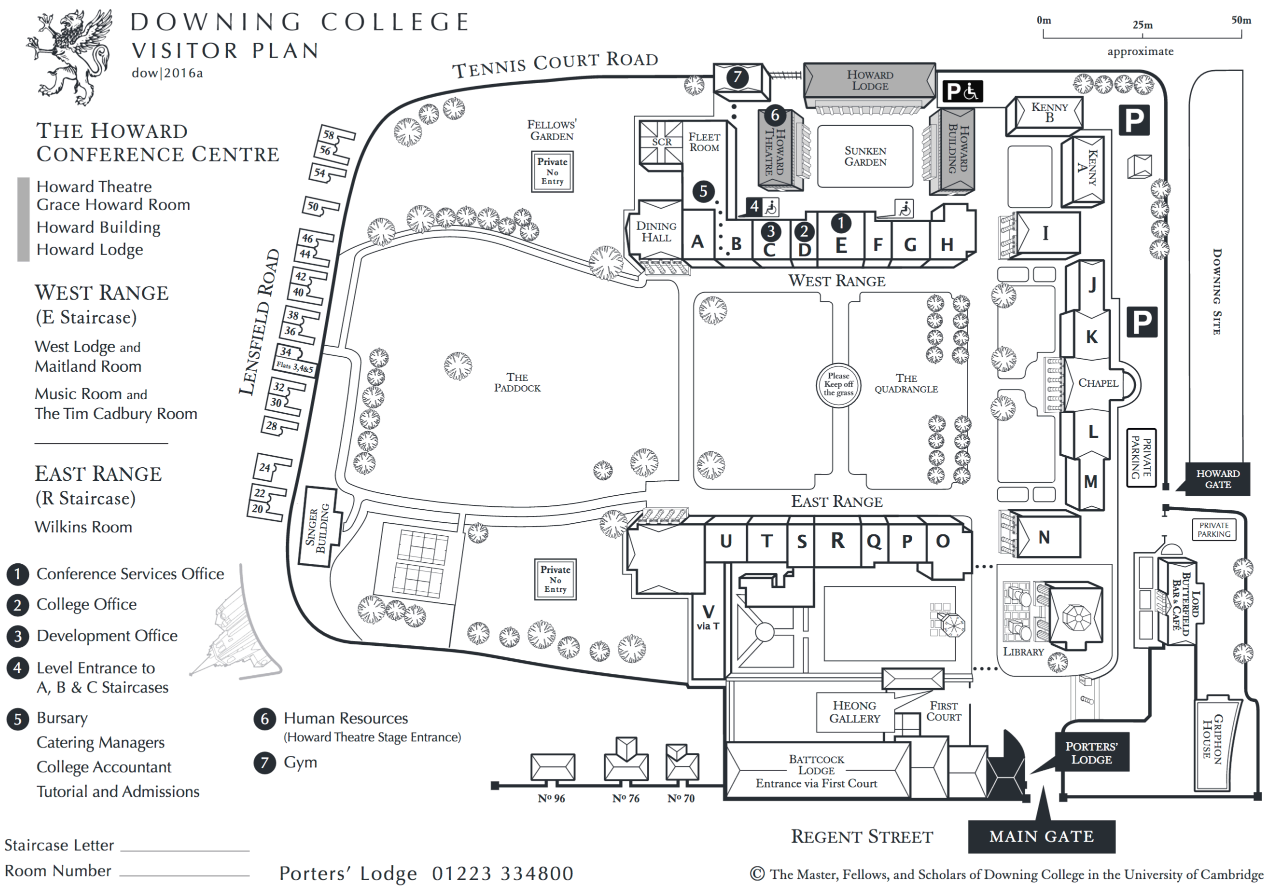


* - Vegetarian § - Gluten-free † - Dairy-free

Dinner Menu

Starter:	Ham hock pressing, grain mustard, apple piccalilli, compressed apple, celery cress, focaccia croute
Main:	Mini-rack and pressed shoulder of lamb, pea and mint puree, crushed parsley potatoes, baby carrots
Dessert:	Baileys crème brûlée, almond shortbread (v)
Veg. Starter:	Gazpacho
Veg. Main:	Shallot tatin, crispy basil tofu, cauliflower couscous, baby vegetables, roast tomato sauce

Plan of Downing College



Abstracts

Session 1 - Materials and Systems in Demanding Industrial Environments

Talk 1: Friday 5th April, 09.00-09.20

Developments in Coating Technology at Westinghouse for Nuclear Applications

DG Eaves

Westinghouse Springfields Fuels Ltd, Station Rd, Preston PR4 0XJ

Westinghouse is currently working on the next generation of nuclear fuels and a number of coating technologies are being investigated. One example is coated cladding to provide enhanced accident tolerance to loss of coolant situations such as that experienced at Fukushima Daiichi following the tsunami in 2011. These coatings may also yield other benefits such as enhanced wear resistance under normal operating conditions. Another area is the application of zirconium diboride to act as an Integral Fuel Burnable Absorber (IFBA) using Plasma Arc Spray. Whilst the IFBA product is well established this coating method offers potential economic and process benefits

Talk 2: Friday 5th April, 09.20-09.40

Integrated Computational Materials Engineering for High Temperatures

RJ Green

Solar Turbines, 2200 Pacific Highway, PO Box 85376, San Diego, CA 92186-5376, USA

Industrial gas turbines require operational flexibility and availability to be successful in the current business environment. Traditionally, operational risk is managed, in part, through scheduled maintenance (based on operation hours), which is typically formulated through a combination of experience and engineering assumptions. These assumptions are usually inherently conservative and therefore limiting to operational flexibility. Customers are often limited to finite operational hours, starts, firing temperatures and/or rotor speeds. Recent advances in physics-based modelling, data analytics and the acquisition of secure machine data have provided a platform for the development of Digital Assets. Digital Assets or twins are created to mirror actual physical assets operating in the field and are a key technology in the implementation of the industrial internet of things (iIOT), enabling operational flexibility and allowing operators to optimize performance, while also maximizing availability. The following presentation provides an overview of the critical elements and modeling approaches needed to successfully build and deploy a functional, efficient Digital Asset and considers the specific material modeling framework.

Talk 3: Friday 5th April, 09.40-10.00

Materials Testing Requirements for Flexible Operation of Gas Turbines

J Douglas

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

Traditional approaches to design, maintenance and overhaul of gas turbines rely on creep life calculations which, inevitably rely on long term, steady state creep data. Materials testing requirements laid out in procedures like ECCC are onerous and expensive and require very long term test programmes over wide ranges of stress and temperature and potentially, multiple batches of material. The introduction of renewable energy to European grids means that gas turbines are subject to much more fatigue. Materials testing requirements have therefore changed. In particular, the interaction between creep and fatigue has become an area of concern for engine manufacturers and owner-operators. A number of cycle types will be presented to show how creep-fatigue interactions can be better understood.

Talk 4: Friday 5th April, 10.00-10.20

Microstructural Evolution in Steam Turbine Components – A Starting Point for More Realistic Lifetime Prediction Methods

T Mayer

MES Institute of Mechanical Systems, ZHAW Zurich University of Applied Sciences
Technikumstrasse 9, CH-8401 Winterthur, Switzerland

In context of the increasingly important role of renewable energy in the European power markets, steam turbines in combined cycle power plants are required to operate more flexibly while still providing a high level of availability and efficiency. Besides the significance of creep loading, steam turbine materials are exposed to low cycle fatigue (LCF) loading at temperatures up to 620 °C. The interaction of creep and fatigue thereby often constitutes a life-limiting factor in steam turbine components that challenges engineers making lifetime predictions both for new and already operating machines. This presentation aims to provide a quantitative insight into the microstructural evolution of a steam turbine rotor steel exposed to LCF at elevated temperatures. Dislocation density and subgrain size evolutions lead to an ongoing softening effect of the material under these loading conditions. A newly developed constitutive model is presented that allows describing this evolution on a more physical basis. This provides the basis for improved methods that allow for more realistic lifetime predictions in critical components.

Talk 5: Friday 5th April, 10.20-10.40

Indentation Plastometry for NDT Mapping and In Situ Monitoring

J Dean, JE Campbell, M Burley & TW Clyne

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

There is serious industrial interest in obtaining bulk materials properties from instrumented indentation data, using iterative FEM simulation of (spherical) indentation and converging on optimal parameter values by optimising the agreement between predicted and measured outcomes. It has already been demonstrated that this is viable for plasticity parameters (such as those in the Ludwik-Hollomon expression). A summary will be given of the current state of the art in these areas. Some attention will be focussed on practical aspects of testing procedures, including optimal indenter size and (relative) penetration levels. These include approaches to in situ testing of components and the potential for using measured residual indent profiles for such fieldwork, during which accurate load-displacement monitoring may be difficult. The overall methodology is becoming increasingly accepted by industry and software packages implementing these capabilities are now starting to become available - see, for example, https://www.plastometrex.com/.

Session 2 - Novel Testing and Investigation Techniques

Talk 6: Friday 5th April, 11.10-11.30

High Resolution Micro-wear Tests of DLC Coatings in a Temperature Range from RT up to 400°C

T Chudoba

ASMEC, Bautzner Landstaße 45, 01454 Radeberg OT Rossendorf, Germany

DLC coatings are widely used in industrial applications due to their high hardness and wear resistance and low friction coefficient. In automotive or other applications, the operational temperature may reach more than 200°C. It is known that the temperature stability of DLC is not very high and a degradation my start above 250 – 300°C. Nevertheless, little is known about the influence of temperature and contact pressure on the friction coefficient and the wear rate. Tribological experiments are mostly done at conditions where the contact diameter is in the mm-range and where the local contact pressure at asperities is unknown. However, the average surface pressure is not a good measure to understand local failures. Micro wear tests with spherical diamond tips of radii between 5µm and 50µm in a nanoindenter with lateral force unit can be used to simulate a single-asperity contact on DLC coatings and to track local friction and wear rate during an oscillatory motion. They gives much better access to failure conditions in dependence on temperature than macroscopic tribological tests. This will be shown for two DLC-coated samples where micro-wear tests have been done with oscillation amplitudes between 4µm and 80µm and at different contact pressures and temperatures between room temperature and 400°C. The measurement of the indentation modulus of the coatings and an accurate calibration of the indenter area function for the spherical tips allowed a precise calculation of the local contact pressure and a derivation of the friction-pressure dependency.

Talk 7: Friday 5th April, 11.30-11.50

Dispersion of Mechanical Properties at Nanoscale studied by Indentation Mapping

UD Hangen[1], E Hintsala[2] & D Stauffer[2]

[1]Bruker Nano GmbH, Dennewartstrasse 25, 52068 Aachen 1

[2]Bruker Nano Surfaces Division, 9625 West 76th Street, Eden Prairie, MN

The mechanical properties of a composite are dependent on the properties of the different components integrated. The size of the components in the composites are ranging from mm down to nm. All components are potentially changing the properties during the production and during the lifetime of the composite. Nanoscale hardness testing has developed significantly in the recent years and the speed of the testing allows now to perform up to 6 measurements per second. These maps can be combined with different environmental conditions such as a temperature range between -120°C and 800°C as well as a humidity range between 5%RH and 95%RH. It is critical for a wider use of temperature dependent experiments to use the instrument time resource efficiently and hence to control the overall cost. The risk of tip changes or sample changes is increasing with increasing time of the experiment. Several strategies are being discussed to allow collecting the spectrum of materials properties as a function of temperature in a faster way. Among other results we will show the hardness and modulus in a SiC-ceramic composite at elevated temperatures as well as the signatures of internal stresses on the local material properties in a semiconductor device.

Talk 8: Friday 5th April, 11.50-12.10

Thermally Activated Dislocation Plasticity in BCC Metals and Alloys studied by High-temperature Nanoindentation

R Schwaiger, C Brandl & I-C Choi
Werkstoffmechanik 1, IAM (Computational Materials Science), Karlsruhe Institute of Technology
Straße am Forum 7, 76131 Karlsruhe, Germany

The application of body centered cubic (BCC) refractory metals is usually limited by the low-temperature brittleness, which is intrinsically linked to the limited screw dislocation mobility. In this study, high-temperature nanoindentation experiments were conducted to determine the dislocation mobility regimes with and without interstitial impurities in pure Cr. Samples having different levels of impurities were tested from room temperature to 600 K and the temperature-dependent hardness, activation volume and apparent activation enthalpy determined. The signatures of the underlying plastic deformation mechanisms are compared to the mobility from theory. A detailed quantitative analysis of the experimental data revealed the solute-drag and kink-pair nucleation-limited dislocation mobility regimes. The latter is subdivided into elastic interaction and line-tension regimes showing characteristic changes of slip system signatures with increasing temperature.

Talk 9: Friday 5th April, 12.10-12.30

Recent Innovation in the Field of In-situ Micro- and Nano-scale Mechanical Testing

NX Randall[1], D Frey[1], Q Longchamp[1], R Ramachandramoorthy[2] , J Breguet[1] & J Michler[1]
[1]Alemnis AG, Feuerwerkerstrasse 39, Thun, CH3602 Switzerland
[2]Laboratory for Mechanics of Materials and Nanostructures, EMPA, Thun, Switzerland

Since emerging in the early 1990s, nano-indentation has established itself as a routine technique for mechanical property measurements, offering a very high degree of flexibility and applicability. It has been used to study the entire spectrum of materials including metals, ceramics, polymers, composites and biomaterials. This talk will introduce in-situ Scanning Electron Microscope (SEM) nano-mechanical testing, which was pioneered by Alemnis AG, showing how the technique has evolved in recent years and how recent innovations have allowed new mechanical properties to be evaluated. Nano-mechanical tests are moving beyond the basic measurement of hardness and elastic modulus to encompass a host of different mechanical properties such as strain rate sensitivity, stress relaxation, creep, and fracture toughness by taking advantage of focused ion beam milled geometries. New developments, such as high cycle fatigue, are extending the range of properties that can be studied at the micro and nano-scale. However, such techniques are challenging due to low oscillation frequencies, long duration of tests and large thermal drift when attempted with standard indentation instruments. Novel piezo-based nano-indentation methods are now allowing access to extremely high strain rates (>10^4 s^{-1}) and high oscillation frequencies (up to 10 kHz). Other recent innovations include cryogenic and high temperature tests covering the temperature envelope from -150 to 800°C. The challenges in variable temperature tests and the associated technological and protocol advances will be discussed along with select case studies. The inherent advantages of using small volumes of sample material, e.g. small ion beam milled pillars, will be discussed, together with the associated instrumentation, technique development, data analysis methodology and experimental protocols. Other complimentary modes, such as scratch testing, will be presented as well as current efforts to combine micro-pillar compression with High Resolution Electron Backscatter Diffraction (HR-EBSD) and Raman scattering. Finally, future research directions in this sub-field of micromechanics will be discussed.

Talk 10: Friday 5th April, 12.30-12.50

Obtaining Miller-Norton Creep Parameters from Indentation Testing

JE Campbell, M Burley, J Dean & TW Clyne

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

Iterative FEM simulation of (spherical) indentation, converging on “solution” combinations of (constitutive law) parameter values by optimizing agreement between predicted and measured outcomes, is now becoming a mainstream procedure, at least for plasticity. Outcomes commonly include load-displacement-time datasets and residual indent shapes. An automated process is required and it must quickly yield accurate and reliable results. This presentation is focused on creep, for which the procedures are less well developed, although the same conceptual approach is used. A key issue concerns the importance of capturing both primary and secondary creep in the constitutive law, since, unlike the case of uniaxial loading, primary creep continues to affect the outcome throughout indentation testing. The experimental outcome is a penetration-time dataset, for a range of different applied loads. Issues related to the modelling include mesh specification, simulation of interfacial friction and options for the convergence algorithm. It is also important to handle the period immediately after indenter-sample contact is initiated, when there is scope for high stresses to be created and plastic deformation to be stimulated. It is shown that, if these various issues are suitably optimized, then reliable Miller-Norton parameter value combinations (consistent with those from conventional uniaxial testing) can be obtained.

Session 3 - Novel Composite Systems for High Temperature

Talk 11: Friday 5th April, 13.50-14.10

Highly Porous Hybrid Particle-Fibre Ceramic Composite Materials for use as Diesel Particulate Filters

AJ Houston & TW Clyne
Dept. of Materials Sci., Univ. of Cambridge, 27 Charles Babbage Road, Cambridge CB3 0FS, UK

This talk concerns an investigation into the potential of a novel type of ceramic composite for use as Diesel Particulate Filters (DPFs). Materials for this application are required to be highly permeable to gas flow, capable of filtering and retaining very fine particulate and be stable under highly demanding thermo-mechanical conditions. Novel materials have been produced, via a conventional blending, cold pressing and sintering route, containing coarse alumina particles and/or fine alumina fibres. A number of relevant thermal and mechanical properties are measured, for a range of fibre contents. Thermal Shock Resistance (TSR) is an important requirement and this has been investigated both experimentally and via evaluation of a TSR merit index, based on a combination of well-defined properties. It is shown that this type of material shows promise in terms of thermo-mechanical stability, particularly when the fibre content is relatively high. There are also indications that such composites would be suitable for DPF usage in terms of permeability levels and the filtration of fine particulate.

Talk 12: Friday 5th April, 14.10-14.30

C3HARME - Next Generation Ceramic Composites for Combustion Harsh Environment and Space

L Silvestroni, L Zoli, A Vinci & D Sciti
CNR-ISTEC, Institute of Sci. & Technol. for Ceramics, Via Granarolo 64, I-48018 Faenza, Italy

The EU-funded project C3HARME aims at combining the best features of CMCs and UHTCs to design, develop, manufacture and qualify a new class of Ultra-High Temperature Ceramic Matrix Composite (UHTCMCs) with self-healing capabilities. Applications selected to implement the new materials are near-zero erosion nozzles and near-zero ablation thermal protection system (TPS) tiles. This presentation introduces the challenges addressed by C3HARME project including i) the integration between well-established and novel techniques for CMCs and UHTCs production, ii) the need for very high temperature characterization, iii) the meaning of self-healing capability for UHTCMCs, iv) the contribution of modelling to materials development and V) the investigation of the microstructure/thermo-mechanical property correlations. Preliminary results concerning production and testing of prototypes in extreme environments are presented too.

Talk 13: Friday 5th April, 14.30-14.50

Ceramic Matrix Composites for Aerospace – Challenges & Opportunities

I Edmonds
Materials Engineering, Rolls Royce, Derby, UK

Rolls-Royce has been developing ceramic matrix composite (CMC) technology for aerospace and power generation gas turbine applications since the 1980s, both internally and externally via academic engagement. Rolls-Royce operates a University Technology Partnership model – as a collective of world-class academic institutions they tackle a wide range of engineering disciplines including OMC, CMC and MMCs. CMCs are seen as a key enabler to improve the specific fuel consumption of aero-engines; research in CMCs is targeted towards two principal application sets – SiC-based materials for core turbomachinery aimed to reduce component cooling flow requirements, and oxide-based materials for weight reduction. The demonstration of technology maturity for introduction into aero-engines is necessarily very rigorous, and the reliability and life requirements for CMCs are particularly challenging for SiC-based high temperature ceramics. These CMCs are expected to operate in a hostile environment combining high temperature, mechanical/thermal stresses, oxidising conditions, a high velocity gas stream and combustion products such as water vapour and SOX. Internal efforts have focussed on design, material and manufacturing capability development. Academic research to develop mechanistic understanding of damage progression, for example, via specialised testing, characterisation of reaction products, and degradation monitoring techniques such as digital image correlation, tomography and acoustic emission, inform the development of lifing methods to underpin component life. This arduous requirement set is also driving parallel activities to develop prime reliant environmental barrier coatings (EBCs) to protect gas path surfaces. For CMCs aimed at weight reduction, typically oxide/oxide CMCs, the challenges are different. Cost reduction, maximising weight reduction via analysis whilst accounting for manufacturing defects, and validating a lifing approach for complex geometries are key. In both CMC types, effectively linking coupon testing to sub-element, sub-component and component testing is challenging, and much reliance on engine testing is inevitable initially. Such is the huge disruptive opportunity of these materials, this step is justified.

Talk 14: Friday 5th April, 14.50-15.10

Challenges during the Fabrication of a Reactive Melt Infiltrated Ultra High Temperature Ceramic Matrix Composite

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

The German Aerospace Center (DLR) is focusing on the development of fiber reinforced Ultra High Temperature Ceramics (UHTC) to enhance the performance of hypersonic flight vehicles. These materials will enable sharp geometries capable of withstanding temperatures above 2000°C, needed in hypersonic engines and leading edges in order to reduce drag. To establish a process for fiber reinforced diborides, DLR investigates a Reactive Melt Infiltration (RMI) of Zr alloys to fabricate carbon fiber reinforced ZrB_2. The purpose of the presented experiments is to further understand the RMI process and increase mechanical performance of the material. Different factors influencing the RMI process are described, in particular the development of a carbon fiber reinforced ZrB_2 by RMI renouncing a protective fiber coating.

Talk 15: Friday 5th April, 15.10-15.30

Understanding Interfacial Failure in SiC/SiC Composites through Micro-mechanical Testing

D Armstrong

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

Silicon carbide ceramics are a candidate material for the use in nuclear power generation and aero engines. This is due to its favorable properties, in particular reduced oxidation under accident conditions, good neutronic performance, high temperature strength and stability under irradiation. Due to its inherent brittleness, it is suggested to be used in the form of SiC-fiber reinforced SiC-matrix composite. In order to reliably model behavior of highly non-uniform and anisotropic composite materials the knowledge of the individual properties of fiber and matrix, and, crucially, the fiber-matrix interfaces, is required. In addition, nuclear fuel cladding materials are exposed to elevated temperatures during their operation, and therefore the understanding of the temperature dependencies of the relevant properties is essential. Micromechanical testing techniques, such as nanoindentation and microcantilever beam fracture, allow determination of such localized properties, and can be implemented in the wide range of temperatures. Here we demonstrate the use of micro-fracture tests, fibre push-out experiments and STEM-EDX to elucidate the controlling chemistry at interface during failure.

Session 4 - Surface Protection

Talk 16: Friday 5th April, 16.00-16.20

Environmental Barrier Coatings for SiC/SiC CMCs: Current Progress in Jülich

E Bakan & R Vassen
Materials Synthesis and Processing (IEK-1), FZJ, 52425 Jülich, Germany

Environmental barrier coatings (EBCs) are developed for advanced gas turbines primarily to eliminate rapid volatilization of hot-section SiC/SiC ceramic matrix composite (CMC) components in the water vapor rich environment as well as to inhibit their oxidation. There are various difficulties with accelerated lifetime testing of EBCs, i.e. to simulate the testing environment with high-velocity water vapor flow and thermal gradient through the sample during thermal cycling. Furthermore, processing of EBCs is known to be complicated due to re-crystallization and secondary phase issues in silicate coatings. In this presentation, the current progress in processing and testing of EBCs will be demonstrated. Characteristics of very low-pressure plasma sprayed and atmospheric plasma sprayed Yb-silicate coatings will be examined. High-velocity water vapor recession (v=100 m/s, P_{O_2}=0.15 atm T=1200 °C) test results of high/low Yb_2SiO_5 containing $Yb_2Si_2O_7$ coatings will be discussed. Finally, thermal gradient cycling tests of thermally sprayed $Yb_2Si_2O_7$/Si EBCs in a modified burner rig test facility will be presented.

Talk 17: Friday 5th April, 16.20-16.40

Hydrogen-metal Interactions by In-situ and Ex-situ Methods

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Understanding hydrogen-microstructure interactions in metallic alloys and composites is a key issue in the prevention of detrimental phenomena such as hydrogen embrittlement. Failure mechanisms initiate at the atomic scale with hydrogen absorption and further interaction with trap binding sites or defects. Nanoindentation and related techniques are valuable tools to study independently such mechanisms due to the small volume probed. Even more, in-situ testing while charging the sample with hydrogen can prevent the formation of concentration gradients due to hydrogen desorption. Two custom electrochemical cells were built for hydrogen charging while nanoindenting the sample for *in-situ* testing: “front-side” charging with sample and indenter tip immersed into the electrolyte, and “back-side” charging where the analyzed region is never in contact with the solution. We discuss the advantages and disadvantages of both approaches during the study of the hydrogen effect on the mechanical behavior and incipient plasticity in bcc FeCr alloys. A reduction in the pop-in load indicating the yield point with the increase of hydrogen content and formation of multiple pop-ins during nanoindentation provided evidence for the decrease in the resolved shear stress and enhanced dislocations nucleation. In additon, an increase on hardness is observed while rising the hydrogen content. *Ex-situ* testing of tungsten is also carried out after introducing deuterium by plasma charging. Tungsten is a proposed material for plasma facing components in fusion experiments. A similar trend is observed as for the in-situ testing of iron alloys. Nanoindentation tests show that exposure to deuterium plasma causes a decrease in pop-in load and an increase in hardness of tungsten in comparison to the unexposed reference sample. Micropillar compression tests demonstrate an increased apparent strain hardening rate as well as an increased multitude of slip traces after exposure. These outcomes are attributed to the presence of deuterium facilitating the dislocation nucleation while may at the same time suppresses the dislocation mobility.

Talk 18: Friday 5th April, 16.40-17.00

Repair of Ni-Superalloys using Thermal Spray

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The hot section parts in aviation engines and stationary gas turbines are exposed to extreme environments, where high temperatures and eroding atmospheres lead to oxidation, corrosion and fatigue damage of the inserted parts. As the manufacture of the nickel-based superalloys for those high temperature applications is expensive, the repair of worn or damaged parts is from an economic viewpoint desirable, however, usually difficult due to the poor weldability of these alloys. The coating technology Cold Gas Spray (CGS) and Vacuum Plasma Spray (VPS) was tested to repair such worn parts and reduce the maintenance cost. In this study Inconel 738 was deposited with CGS on polycrystalline Inconel 738 substrates. Additional heat treatments were performed to investigate the microstructural change. Indentations were carried out at elevated temperatures (850 °C) to simulate the stress-strain curves of two different repair approaches. The comparison of those results to the substrate shows the great potential of this technique. VPS was used for the repair of single crystalline CMSX-4 substrates. An additional heat treatment with a hot isostatic press led to dense polycrystalline repair coatings. The stress-strain curves of those coatings were simulated after indentation and the results were compared to the microstructural features of the coatings. A new process will be introduced for the future single-crystalline repair of VPS coatings.