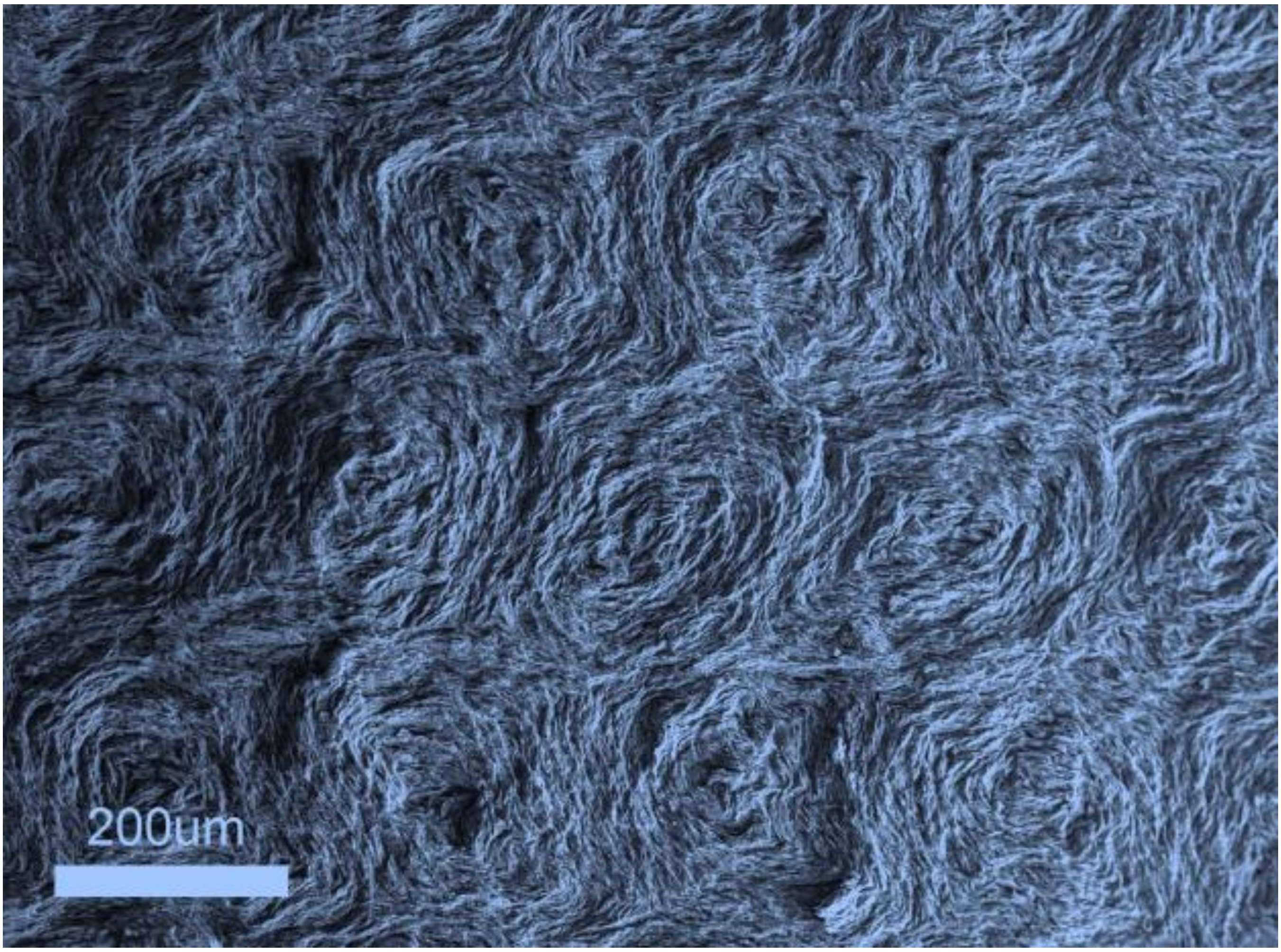
Centre for Advanced Structural Ceramics
Annual Report – 2015



Contents

Introduction

CASC started in July 2008 with EPSRC funding (£5.5M) for a five-year programme. The EPSRC funding came to an end in June 2013, but CASC continues to grow, establishing a large number of active industrial collaborations from abroad and in the UK.

An Industrial consortium has been set up to build on CASC's early success, enable its sustainability and to continue long-term and fruitful relationships between CASC's associated academics and the UK's ceramics community.

Prof Eduardo Saiz
CASC Director

Management

CASC was initially set up by Professor Bill Lee in 2008. Professor Eduardo Saiz took over as Director in 2012.

Local management team (LMT)

The LMT is responsible for managing the centre's operations and meets monthly to oversee the pressing day-to-day issues of running the Centre. These issues include staff appointments, equipment purchases and building refurbishment, but are increasingly focussed on developing the Centres national and international profile, forging industrial links and achieving financial sustainability. The LMT is chaired by Eduardo Saiz and other members are Dan Balint, Finn Giuliani, Julian Jones, Kamran Nikbin, Luc Vandeperre, Stephen Skinner, Garry Stakalls, and Amutha Devaraj. LMT meetings are also attended by two representatives of the postdoctoral researchers (Victoria G. Rocha) and PhD students (Omar Cedillos Barraza) working on projects related to structural ceramics.

Industrial Consortium Group (ICG)

A key part of CASC's sustainability is the development of a consortium of companies with interest in structural ceramics. To build on CASC's early success, to enable its sustainability after the EPSRC funding ends in 2013, and to build long-term and fruitful relationships between CASC-associated academics and the UK's industrial structural ceramics community we have set up an industry consortium scheme. These plans were presented to our first Industry Day meeting on 17 May 2011, where they were well received by the industry representatives and were developed by our Steering Group on 4 July 2011. The industrial consortium started functioning from 2014 after the steering group meeting held 17 January 2014. The ICG develops the CASC Business Plan which contains the Centre vision, objectives and an action plan to deliver the vision. It acts in an advisory role to the Director and to the Local Management Team, in particular providing advice on:

a. The strategic research focus of the Centre
b. The infrastructure, skills needs and links to industry and other research groups worldwide
c. The structure and content of undergraduate and postgraduate courses provided by the Centre

The consortium has three levels of membership with a graduated annual fee and access to CASC facilities, people and projects as shown in the table below.

Diamond membership is aimed at large and multinational companies, who would like strategic advice and board-level interaction with senior academic staff at CASC. The relationship, which might include technical briefings and RAEng Industrial Fellowships, would be tailored to individual company requirements.

Sapphire and ruby memberships are aimed at companies who want to collaborate with CASC on research and training; sapphire membership offers greater benefits that ruby membership.

All three levels of membership provide:

- Access to CASC equipment (including hot press, vacuum furnace, nanoindenter etc.) and Department of Materials central facilities (includes X-ray Diffraction, Electron Microscopy, Secondary Ion Mass Spectroscopy and Thermal Analysis), with operator and interpretation. Access to our faci

- lities is at preferential rates – much reduced compared to the ones for outside users. The degree of access will depend on the level of membership.
- Access to CASC and CASC associated academics.
- Positions in the CASC Ceramic Summer School
- Access to Materials, Mech. Eng. and CASC students as potential employees.
- Opportunity for secondment of industrial researchers to CASC.
- Opportunity to propose undergraduate final year research projects, at differing levels depending on membership. Projects run from October to May, descriptions needed by Easter previous year.
- Opportunity to propose research projects for students on Masters Courses (Advanced Materials, Biomaterials & Nuclear), at differing levels depending on membership. Projects run from April to September, descriptions needed by May previous year.
- Opportunity to collaborate on out-of-term and industrial placements. Interviews can take place from October onwards.
- Receiving the CASC annual report and newsletter as well as information on CASC sponsored events
- Opportunity to propose a PhD consortium studentship subject. Members will have access to results and analysis resulting from the three year project.
- To date we have five members signed up at Sapphire level (Morgan Advanced Materials, DSTL Rolls-Royce, Kerneos and Asahi Glass) and one member at Ruby level (Reaction Engines) and are in advanced discussions with several other companies. If you are interested in becoming a member of the CASC Industry Consortium, contact: Eduardo Saiz – e.saiz@imperial.ac.uk - 020 7594 6779 / Amutha Devaraj – amutha.devaraj@imperial.ac.uk – 020 7594 2053

	Diamond	**Sapphire**	**Ruby**
Type of membership	Strategic	Research & Training	Research & Training
Steering Group member	Yes	Yes	Yes
Equipment use **Free allowance up to** **Preferential rates for other use**	£10,000 Yes	£3,000 Yes	No Yes
Proposing MSc, BEng, and MEng projects	8	2	1
Access to CV's of graduating students	Yes	Yes	Yes
Free summer school positions	10	3	1
Membership fee	£50,000+VAT	£15,000+VAT	£5,000+VAT

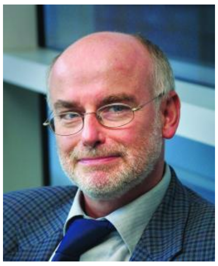
People

Staff

Professor Eduardo Saiz

CASC Director since August 2012. Eduardo Saiz, previously a Staff Scientist at the Materials Sciences Division of Lawrence Berkeley National Laboratory (LBNL), joined CASC in October 2009. Eduardo took over the role of Deputy CASC Director in July 2010. He has been the Vice-chair of the Basic Science Division of the American Ceramic Society.

After graduating in Physics from Cantabria University in Spain he gained a PhD in Applied Physics from the Autonoma University of Madrid, working on the processing of ceramic superconductor thick films. In 1992 he became a Fulbright postdoctoral researcher at LBNL. He has worked extensively in the area of high-temperature capillarity and interfaces between dissimilar materials, developing new approaches to study spreading and adhesion in metal-ceramic systems – this continues to be a topic of research. Another area of interest is in the development of new hierarchical, hybrid materials and coatings (metal/ceramic, polymer/ceramic) as well as complex porous ceramics. One of the objectives is to develop high-temperature composites able to perform in extremely hostile conditions and increase efficiency in the transport and generation of energy. He is also working in the fields of biomineralization and the development of new ceramic-based biomaterials to enhance the osseointegration of orthopaedic implants and support the engineering of new bone and cartilage.



Professor Bill Lee

Professor Bill Lee was the founding Director of CASC from July 2008 until August 2012 and is the principal investigator of the EPSRC award. Bill is a Professor of Ceramic Engineering and was Head of the Department of Materials at Imperial College London from January 2006 to August 2010. After graduating in Physical Metallurgy from Aston University he gained a DPhil from Oxford University on radiation damage in sapphire, was a post-doc at Oxford and Case Western Reserve Universities, Assistant Professor at Ohio State University, USA before becoming lecturer in ceramics at the University of Sheffield in 1989. While at Sheffield he was Manager of the Sorby Centre for Electron Microscopy and Director of the BNFL university research alliance the Immobilisation Science Laboratory. Bill was made a Fellow of the Royal Academy of Engineering in 2012 and his current research is focussed on ultra-high temperature ceramics for aerospace applications and ceramics for nuclear fuel and waste immobilisation applications.

Dr Finn Giuliani

Dr Finn Giuliani joined us in April 2009 as a lecturer joint between the Departments of Materials and Mechanical Engineering. Finn came to Imperial from Linköping University, Sweden where he was an Assistant Professor following.

Finn has a PhD from the University of Cambridge where he examined small scale plasticity in multilayered ceramics coatings. Particular emphasis was placed on measuring and observing small scale plasticity at elevated temperatures. His BEng in Materials Science and Engineering is from the University of Bath. While in Sweden he concentrated on deformation of a group of nanolaminated ceramics known as MAX phases. These are a group of ternary nitrides and carbides, for examples Ti_3SiC_2, which combine ceramic and metallic properties. However, of particular interest is their ability to dissipate energy through reverse plasticity. This continues to be a topic of research. He also has interest in ternary nitride systems which offer the possibility of an age hardenable ceramic. These systems are of particular importance to the cutting tool industry. He also has new projects starting in the area of boron carbide for amour applications.

Finally, he has an interest in novel *in situ* mechanical testing regimes whether in TEM, SEM or synchrotron.

Dr Luc Vandeperre

Dr Luc Vandeperre, currently a senior lecturer in the Department of Materials, joined the CASC academic staff on 16 July 2010 Luc joined Imperial College in 2006 from the University of Cambridge, where he was a post-doctoral research associate. During his PhD at the Catholic University of Leuven (Belgium), he investigated the electrophoretic deposition of layered ceramic shapes, and was awarded the 1997 Scientific Prize of the Belgian Ceramic Society for his work. Since then, he has worked in both commercial as well as academic environments researching the shaping of ceramics and understanding their thermo-mechanical properties. In addition to his PhD research he has carried out research on shaping ceramics and ceramic foams using natural binders such as starch and gelatine, thermal shock of ceramics, fracture of laminated ceramics, fracture of porous brittle materials, and the relation between hardness and deformation mechanisms. He also designed a device capable of thermal compensation of fibre Bragg gratings for optical data transmission.

Dr Vandeperre's current research spans two themes. The first is thermomechanical properties of structural ceramics, where he is investigating ceramics for use in high temperature environments and as ballistic protection. A second theme is environmental technologies. In this area, he is involved in research into cements for nuclear waste encapsulation, tailoring materials for anion removal from water and producing high value products from industrial by-products.

Dr Amutha Devaraj

Amutha joined the Department of Materials as Technical Manager in April 2013. Prior to this she worked as a Team Leader (Quality and Materials) at Novacem, a carbon negative sustainable material development company. She is involved in technical and administrative activities of Centre for Advanced Structural ceramics (CASC). She has experience working on the development of wide range of materials including ceramics, glass and polymer for industrial applications. She also engages herself in the BioBone (European FP7 project) and Programme Grant (XMat, EPSRC) within the Department of Materials.

Garry Stakalls

Garry Stakalls started as technician for the Centre in July 2008. Prior to this he worked in the Materials Processing Group within the Department of Materials, where he commissioned and ran large experimental rigs and was involved in the processing of wide range of materials. His main activities have been to use and train new users on the use of the thermal analysis equipment as well as operating the hot press for sintering and pressing. He also maintains the equipment while liaising with Netzsch for thermal analysis and FCT for the hot press.

Researchers

Dr Doni Daniel	Research Fellow
Dr Victoria G. Rocha	Marie Curie Fellow
Dr Na Ni	EPSRC Junior Research Fellow
Dr Esther García-Tuñón	Research Associate
Dr Daniel Glymond	Research Associate
Dr Laura Larrimbe	Research Associate
Dr Ayan Bhowmik	Research Associate
Dr Nasrin Al Nasiri	Research Associate
Dr Eleonora D'Elia	Research Associate
Dr William Montague	Research Associate
Dr Vatish Patel	Research Associate

PhD students

Jennifer Alex	Ahmad, Nor Ezzaty
Edoardo Giorgi	Yun-Hao Hsieh
Zoltan Hiezl	Wirat Lerdprom
Claudio Ferraro	Giorgio Sernicola
Gil Machado	Tom Giovannini
Annalisa Neri	Ezra Feilden-Irving
Cyril Besnard	Alan Leong
Tommaso Giovannini	Claudia Gasparrini
Omar Cedillos Barraza	

Sabbaticals and visitors

Prof Theodre Bessmann	University of South Carolina
Prof Michel Barsoum	Drexel University, Philadelphia, USA
Dr. Carolina Clausell Teroll,	Universitat Jaume I, Castellon, Spain
Mr. Massimiliano Dapporto,	National Research Council of Italy, Faenza (Italy)
Dr. Maria Cabanas Corrales,	Acerlor Mittal, Spain
Dr. Manuel Miranda Martinez	ITMA, Span
Dr. Antonio Paez Duenas	REPSOL, Spain
Dr. Andy Goodwin	Thomas Swan, Spain
Prof. Dr. Andre R. Studart,	ETH Zurich, Switzerland

Dr. Alaitz Rekondo & Dr. Ibon Odriozola,	IK4 CIDETEC, Spain
Mr. Chris Primett	Welland Medical, UK
Dr. Arash Moavenian	Welland Medical, UK
Mr. John M. Finley II,	MEMPRO Materials, USA
Dr. Tran, Hwee Nah Serena & Dr. Goh, Joo Thiam and Ms Teo, Jia Hui	DSO National Laboratories Singapore
Dr. Alan Porporati	Ceramtec, Germany
Dr Leong Kok Hoong	Petronas, Malaysia

CASC Alumni

Suelen Barg	Acad. Staff at Univ. of Manchester (in process)
Miriam Miranda	Element six
Salvador Eslava	Acad. Staff at University of Bath
Na Ni	EPSRC Junior fellow, ICL
Amanda Quadling	Morgan Advanced Materials
William Montague	PDRA CASC (Luc Vandeperre)
Nasrin Al Nasiri	PDRA CASC (Bill Lee)
Eleonora D'Elia	PDRA CASC (Eduardo Saiz)
John Mitchell	Nextec Ltd.
Daniel Glymond	PDRA CASC (Luc Vandeperre)
Rui Hao	PDRA, University of Illinois
Vineet Bhakhri	PDRA University of Western Ontario
Jianye Wang	John Crane, UK
Philip Howie	Cambridge - PhD
Janke Ye (Sheffield)	submitting thesis in January
Ben Milsom (QMUL)	QMUL - PhD
Claudia Walter (IBM)	IBM
Mr Constantin Curea	Rolls-Royce
Bai Cui	Assistant Professor, Nebraska-Licoln University, USA
Naeem Ur-rehman	PDRA KAUST and now Baker Hughes, SA

Research

PDRA Projects

High Emissivity Coating to Improve Thermal Efficiency of Steam Cracking Furnaces

Researcher : Doni J. Daniel
Supervisor : Bill Lee
Sponsors : SCG Chemicals Ltd., Thailand

Petrochemical industries have offered significant contributions to the society through their various end-products, ranging from cosmetics, fertilizers, plastics, asphalt and gasoline. Such industries are currently looking for new advanced technologies to reduce the fuel cost. Besides developing new alternative advanced cracking technologies, the development of new materials for coils and cracking furnaces has become an alternative method to improve the thermal efficiency of furnaces. An increase in the efficiency of heat transfer within the system leads to a reduction in fuel consumption and an increase in yield of products.

High emissivity coating has been widely used in pyrolysis section of steam cracking furnace to enable an effective heat transfer. A high emissivity material is applied to cover the inner surface of furnace wall, enabling the surface to absorb and remit the heat received. It has been reported that this technique is capable to increase at least 5% of thermal efficiency for the same amount of heat input (Heynderickx & Nozawa, 2004). The typical material applied as high emissivity coating in steam cracking furnace is chromium oxide, which is operated at 1100°C.

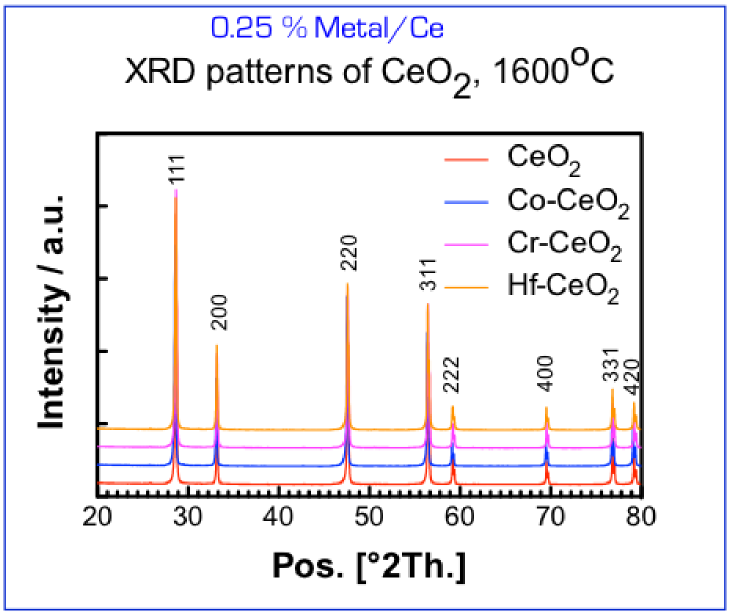
Meanwhile, it is believed that an increase in operating temperature enables the reaction to occur more spontaneously and rapidly as the excess heat provides more energy to overcome energy barrier. As a result, more products can be obtained. The petrochemical industries are currently aiming to enable the reaction in the pyrolysis section at 1600°C. However, chromium oxide, which is currently being used, can only withstand the operating temperature at 1100° or below. Above that temperature, the high vapour pressure of chromium causes the coating material to become unstable and start to disintegrate, leaving the rest to melt. Similarly, other 3d-metal oxide systems have high vaporisation properties, particularly at high temperature, causing them to become non-resistant to reactive atmosphere.

Cerium oxide (CeO_2) is a rare earth based ceramic material that is well-known for its refractoriness, having a melting point of 2600°C. This ceramic is also highly stable and has a good thermal resistance, making it suitable for high temperature applications in an extreme environment. It is stable at high temperature, and the high emissivity characteristic of cerium oxide can be applied from 1000°C to 2000°C, without any degradation. However, due to its high refractoriness, one challenge of applying CeO_2-based material is its manufacturability, mainly the sinterability. Several studies reported that even at high temperature, above 1500°C, pure CeO_2 cannot achieve full density. In addition, further increase in sintering temperatures can lead to excessive grain growth, causing the mechanical properties of material to deteriorate.

The objectives of this project are three-fold: (i) To improve the sinterability of cerium oxide, (ii) To develop a multi-layered coating technique to improve the emissivity at high temperature and (iii) To develop a technique to measure thermal emissivity at high temperature.

(i) Improving the sinterability of cerium oxide

CeO_2 was doped with different dopants and were sintered at 1600C for 3h. Figure 1 shows the XRD patterns of sintered CeO_2 doped with different dopants. No traces of secondary phases were observed and the lattice parameters were calculated from XRD peaks and given.



	lattice param (Ang.)
Pure CeO_2	5.4115(9)
Co - CeO_2	5.4109(8)
Cr - CeO_2	5.4110(9)
Hf - CeO_2	5.4109(9)
Y - CeO_2	5.4107(9)

No significant difference in lattice parameters was observed.

No trace of secondary phases were observed.

Figure 1 XRD patterns of doped CeO_2 and the calculated lattice parameters.

The table below shows the sintering conditions and results of CeO2 doped with different dopants. Dopants significantly reduced the grain growth of pure CeO2 (30- 40 m) to less than a micron.

	Atomic ratio	Sintering Temperature °C	Relative Density %	Grain size µm	Remarks
CeO_2		1600	94	30 - 40	Exagerrated grain growth
Y - CeO_2	0.25	1600	96	2 - 4 & 0.5 - 1	Bimodal distribution
Y - CeO_2	1.00	1600	93	2 - 4 & 0.5 - 1	Bimodal distribution
Yb - CeO_2	1	1600	99	1.00 - 3.00	
Co - CeO_2	0.25	1600	97	6.00 - 10.00	Surface corrosion
Cr – CeO_2	0.25	1600	82	~ 5.00	Needle-like grains

The microstructures of sintered CeO2 were shown in Figure 2.

This research study will offer a potential benefit for petrochemical industries. By developing a coating material that is capable to withstand at higher temperature, the process of steam cracking might be operated at higher temperature. This could provide a faster process, providing higher yields, which is favourable for the companies. Furthermore, the application of high emissivity coating in steam cracking furnace could reduce the fuel consumption, leading to energy saving and a decrease in production cost.

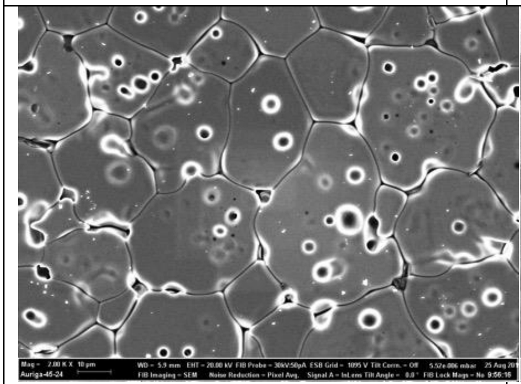
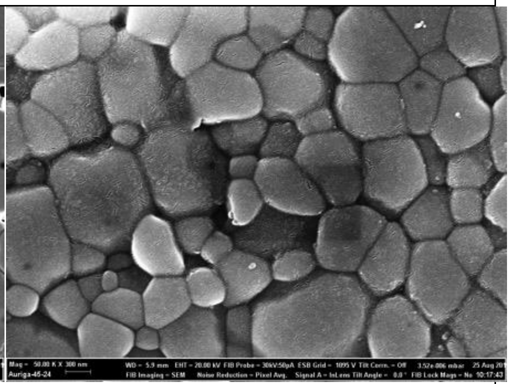
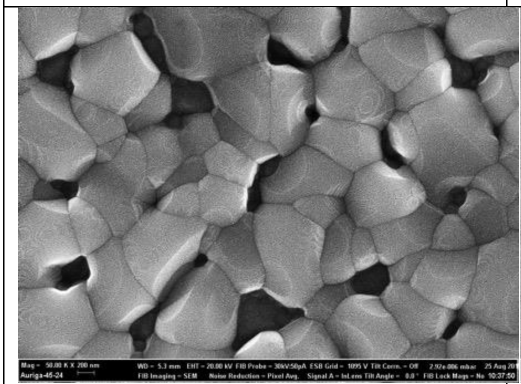
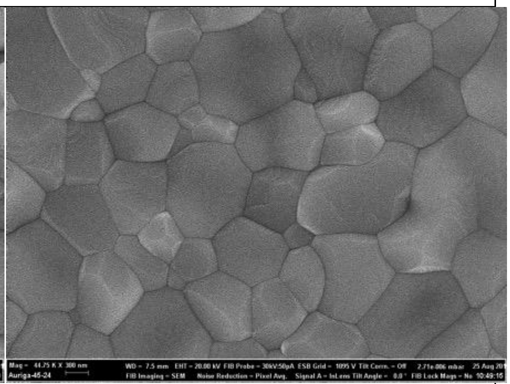
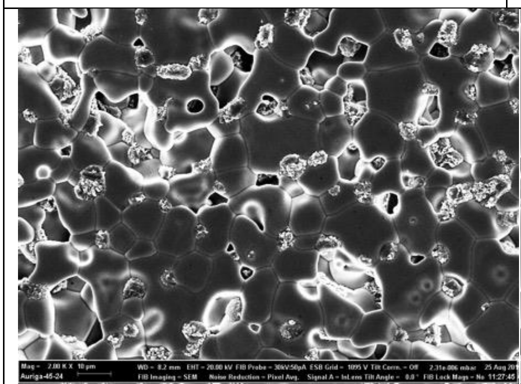
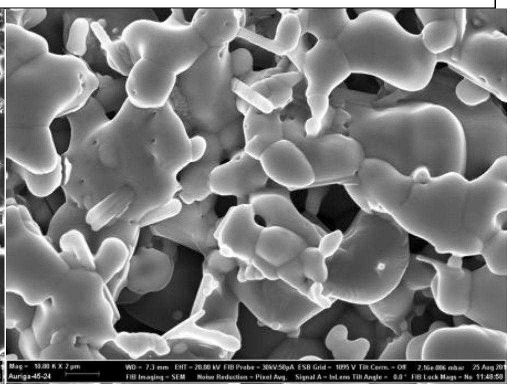
Pure CeO_2	Y/Ce=0.25 atomic ratio
	
Y/Ce=1 atomic ratio	Yb/Ce=1 atomic ratio
	
Co/Ce=0.25 atomic ratio	Cr/Ce=0.25 atomic ratio
	

Figure 2 Microstructures of CeO_2 doped with different dopants

Graphene Enhancement of the Photocatalytic Activity of Semiconductors

Researcher : Victoria Garcia Rocha
Supervisor : Eduardo Saiz
Sponsor : Marie Curie Intra-European Fellowships

The ultimate goal of my research is the development and manufacture of a new family of supported photocatalyst materials, based on graphene, which are active under a broad range of the solar radiance due to their added effectiveness under visible light. The achievement of this goal would allow the direct implementation of these materials on real applications as self-cleaning surfaces, water splitting, and air/water purification.

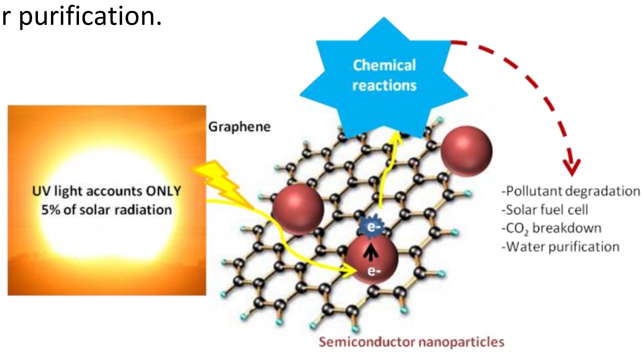


Figure 1 Schematic view of the hybrid photocatalyst working

Other research topics are related to the semi-continuous production of graphene oxide or the new approaches of processing and developing graphene/ceramic composites for energy storage applications or structural ceramics.

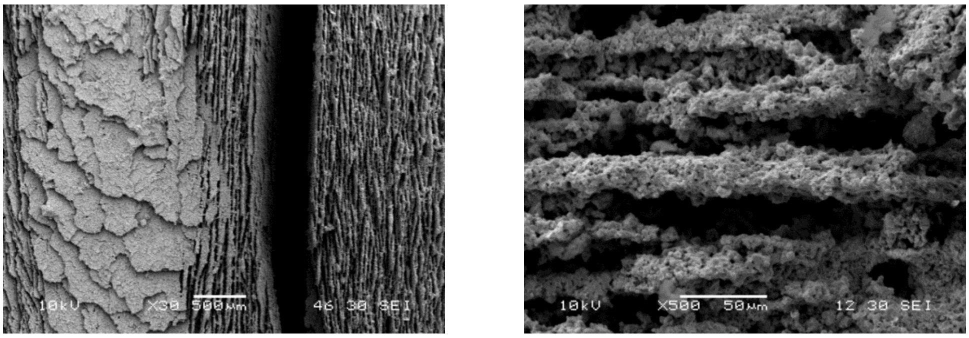


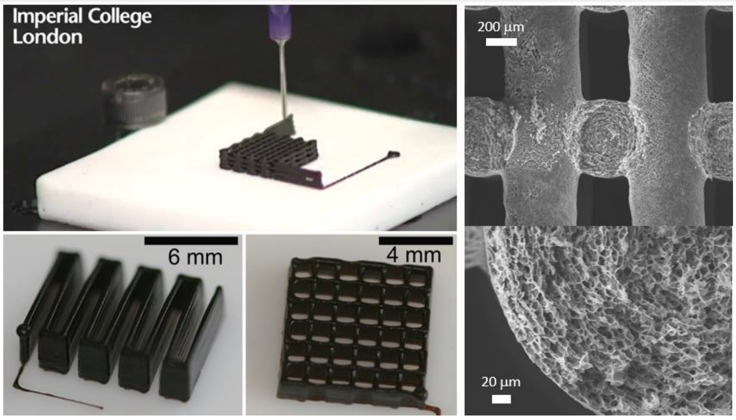
Figure 2 SEM images of freeze casted structures of $LiFePO_4$

Graphene 3D networks

Researcher :Esther Garcia-Tunon,
Supervisor :Eduardo Saiz
Sponsor : EPSRC

Graphene is a fascinating material with a unique combination of properties. But to actually exploit all its advantages, it is necessary to develop manufacturing routes to create macroscopic three-dimensional parts, to integrate graphene into practical devices. We use graphene oxide (GO) as building block and direct ink writing (DIW) to create macroscopic GO 3D structures. This technique is based on the continuous deposition of a filament following a computer design. The 3D structures are built layer by layer from bottom to top.

We have developed a new processing approach to design responsive graphene inks for direct write assembly. We use different responsive molecules (pH and temperature) to functionalize the surface of graphene oxide (GO) flakes to create ‘smart’ building blocks. These ‘smart’ building blocks can be assembled ‘on demand’ in a suspension by using an external stimulus (pH or temperature). This binds the GO flakes through hydrogen-bonding forming a network that provides a wide range of soft-materials that can be used in different processing approaches. From molding, tape casting and



emulsion templating, to DIW. Once the structures are built, we recover the multifunctional properties of reduced graphene oxide after freeze-drying and thermal reduction. We are now studying their application in electrodes for supercapacitors and pressure sensors.

Figure. Printing GO, examples of wet structures and internal microstructure after reduction.

Studying micromechanics of deformation in complex materials using combination of micropillar and *in-situ* micro Laue techniques

Researcher : Ayan Bhowmik
Supervisor : Finn Giuliani
Sponsor : EPSRC

Understanding the response of brittle materials, like ceramics and intermetallics, under loading plays a crucial role in the designing of engineering components out of such materials. This work investigates the micro-mechanics of such materials and attempts to capture the processes that are operative during the initial stages of plastic deformation before the material ultimately fails essentially by brittle fracture. To measure this we have carried out white beam micro Laue diffraction while micropillar compression *in situ* within synchrotron facilities in Diamond* and Advanced Photon Source†. This provides a unique protocol to study the fundamental operating mechanisms of deformation of material in small scale. We have independently characterised the deformation behaviour of two single crystal micropillars: (a) [100]-oriented MgO, and (b) [110]-oriented complex $Al_{13}Co_4$ intermetallic. By tracking the movement and streaking of the Laue reflections, a significant amount of information about the dynamics of underlying deformation mechanisms in materials can be obtained. In MgO micropillars, plastic strain observed will be compared with crystal plasticity modelling. The direction of streaking of peaks in $Al_{13}Co_4$ was found to be consistent with the activity of slip on the (001) plane of the crystal that results in the generation of high density of dislocations and planar faults. In future, efforts would be made to investigate various other structural alloys to understand how they behave under applied stresses in operating conditions.

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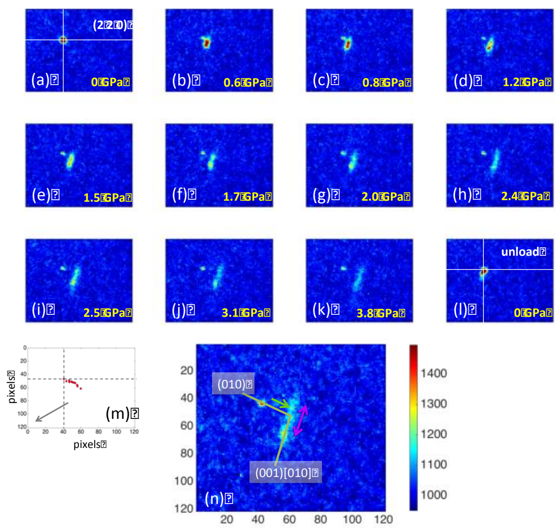


(a-l) Evolution of the (220) reflection with increasing compressive stress during in-situ straining of $Al_{13}Co_4$ micropillar showing rotation and streaking of the peak. (m) Legend showing the movement of the peak centres with increasing strain. The arrow points to the direction of the transmitted beam. (n) The streaked reflection on which is superimposed the vectors denoting the direction of rotation and streaking due to the operation of two different slip systems in $Al_{13}Co_4$.

Diamond Light Source, Rutherford Appleton Laboratory, Didcot, UK
† Advanced Photon Source, Argonne National Laboratory, Argonne, Chicago, USA

Ceramic materials and shaping technologies for short life propulsion systems

Researcher : Daniel Glymond
Supervisor : Luc Vandeperre
Sponsor : Microturbo

Powder pressing is industrially established but not well suited to the production of complex shaped turbine blades and components. However, pressing followed by green machining is applied successfully in space components and could potentially offer a way to produce intricate shapes while taking advantage of the well-developed pressing technology. Alternative shaping methodologies find their origin in clay based ceramics and are based on suspensions of ceramic powders: wet processing or colloidal techniques. A wide range of variants, often differing only in a limited way, exist. From these a number appear promising. In gelcasting a gel former is added to the ceramic suspension so that it can be cast and subsequently gelled in a mould. The shape can then be demoulded, dried and sintered. Omatate from the Oak Ridge National Laboratory in the USA developed this methodology and used a radial-vane turbine rotor made from silicon nitride as a key example component[1]. However, drying of gelcast components can be cumbersome especially if the dimensions increase. Nevertheless, it seems obvious that this methodology must be part of the mix. In later years, the use of binders from the food industry such as gelatine have been investigated and these have clear advantages over the polymer monomers in terms of toxicity[2]. A family of similar techniques are termed coagulation casting, in which a fluid suspension is made to solidify by removing the interparticle repulsive forces (for example by an homogeneous change in the suspension pH) once the object has been cast. An alternative is to engineer responsive particle surfaces that will promote particle dispersion in a suspension and subsequent assembly in response of an external trigger (e.g. pH). This could be done, for example, by grafting the particle surfaces with a pH responsive polymer such as a BCS polymer.
Finally, another way to produce ceramic parts with complex shapes is the use of solid-free-form fabrication techniques in which the part is printed in three dimensions following a computer design. We will focus on robotic assisted deposition, a continuous extrusion technique that can be used to print a sample layer by layer employing colloidal ceramic inks. The inks have to be pseudoplastic so they flow in the printing nozzle under pressure and after printing they can sustain the weight of the part without deformation. This can be achieved by, for example, using inks based on thermally reversible hydrogels that are fluid at low temperature allowing the homogeneous dispersion of ceramic particles and pseudoplastic at room temperature, allowing printing.

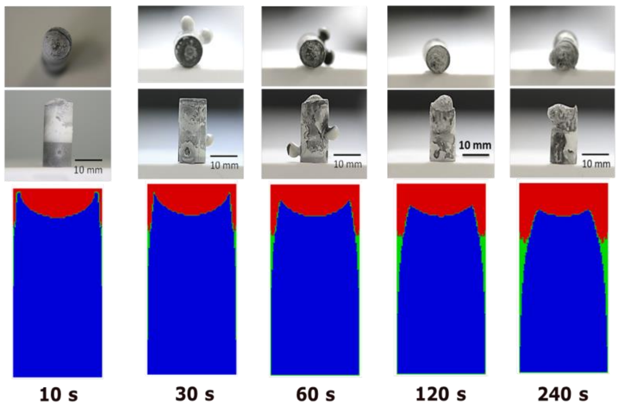
- Janney, M.A., et al., *Development of Low-Toxicity Gelcasting Systems.* Journal of the American Ceramic Society, 1998. **81**(3): p. 581-91.
- Vandeperre, L.J., A.M. De Wilde, and J. Luyten, *Gelatin gelcasting of ceramic components.* Journal of Materials Processing Technology, 2003. **135**: p. 312-316.

High heat flux laser testing of HfB_2 cylinders

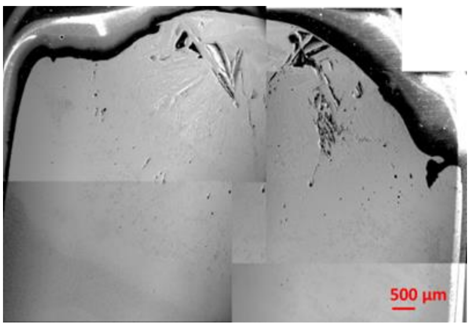
Researcher : Laura Larrimbe
Supervisor : Luc Vandeperre
Sponsor : DSTL (Defence Science and Technology Laboratory)

Hafnium diboride (HfB_2) is one of a family of ultra-high temperature ceramics (UHTCs) which is being considered for application in environments with a substantial heat flux such as hypersonic flight due to the combination of properties it offers, such as high melting point, good oxidation resistance, excellent thermal conductivity, high strength and hardness and good chemical stability. To evaluate the performance of UHTCs, some of the most important criteria are thermal stress and oxidation resistance. Actually, oxidation under hypersonic flow conditions and environmental degradation are extensively recognized as key life-limiting factors for leading-edge application. Oxidation changes the bulk and surface properties of UHTC materials, as oxides have lower thermal conductivities and lower emittances than virgin UHTC materials, and consequently, are less able to dissipate heat by conduction into the interior or radiation back to the environment.

Laser testing is a reasonable cost method for predicting the in-service behaviour of HfB_2 based compounds that uses a laser as the heating source to produce the desired heat flux. In this work, in order to characterise transitions in the material response with heat flux and therefore predict the in-service behaviour of UHTCs, a range of tests were conducted in which small cylindrical bars of HfB_2 were laser heated using heat fluxes from 25 to 100 MWm^{-2}. After testing, the external damage as well as damage observable in cross-sections through the cylinders was characterised using photography, optical, and scanning electron microscopy (SEM). Experimental results were compared with finite element (FE) modelling of the heat flow, temperature distribution and phase transition. Heat flux rather than total deposited heat was found to be the strongest determinant of the way in which damage develops in samples; for lower heat fluxes the main damage mechanism is oxidation, progressing to oxidation induced melting and finally, at the highest heat fluxes, substantial ablation by melting irrespective of oxidation. The agreement between calculations and experimental observations indicates that such calculations can be used with confidence to guide the design of components. While the different stages in damage formation can be expected to occur for all sample sizes, the sample shape is very important in determining the actual values of the heat flux for damage regime transitions. This shows the importance of relatively straightforward modelling to be carried out in parallel with experiments to aid the interpretation of the results and to build confidence in predictability of the damage for other circumstances.



(a)



(b)

Figure 1 (a) Photographs of the samples tested at 50 $\mathbf{MW\ m^{-2}}$ for different times as well as predicted damage at the end of the dwell at maximum powder where red is molten material, green is oxidation and blue is unaltered HfB2; (b) SEM micrograph of the cross section of the sample exposed to 50 $\mathbf{MW\ m^{-2}}$ for 60s.

Irradiation and Thermal Damage of Tungsten Carbide

Researcher: Sam Humphry-Baker
Supervisor: Bill Lee
Sponsor: EPSRC (XMat grant), Tokamak Energy

The development of advanced structural and armour materials is a pressing challenge for compact nuclear fusion reactors. This project is focussed on developing tungsten carbide composites for such reactors, where recent computational studies show they can be efficient for shielding high-energy neutrons. Three work-streams were undertaken: (i) baseline oxidation studies; (ii) development of self-passivating structures; and (iii) helium ion irradiation.

Oxidation tolerance is key design requirement for accident safety in a fusion reactor; in the event of a loss of coolant event, oxidation could release volatile tungsten oxide vapours and transmutation products. The oxidation kinetics of tungsten carbide were systematically investigated and compared to conventional candidate materials (Fig. 1). We found a considerable discrepancy with previous studies, which was explained by high levels of porosity in such works. Our detailed structural characterisation of the oxide films revealed that a protective sub-stoichiometric layer controls the oxidation rate – and that high levels of porosity would likely negate its protective role.

To enhance the resistance of these materials to oxidation, self-passivating structures were developed. WC-Fe composites were diffusion impregnated, and a decrease in the oxidation rate constants by 3-4 orders of magnitude was achieved, which dramatically outperforms conventional boriding treatments. The performance enhancement is explained by the formation of an outer layer that was rich in Fe, and poor in W, which resulted in selective oxidation of the impregnated species.

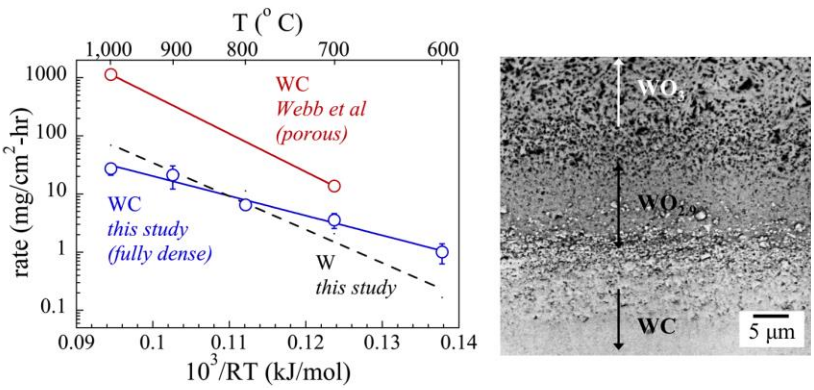


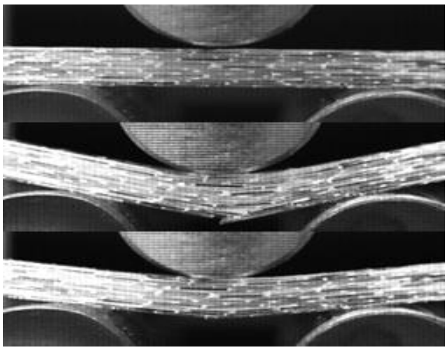
Fig. 1 Oxidation rate constants (left) and SEM image of the oxide film (right).

Finally, helium gas is produced in reactor materials via transmutation – particularly in carbon based materials. Such ions tend to induce bubble formation, which can degrade mechanical and thermal properties. Implantations were performed on TEM foils at the Microscope and Ion-Accelerator for Materials Investigations (MIAMI) at the University of Huddersfield, and monitored in-situ. Irradiations were performed at 6 keV to a total dose on the order of 10^{17} ions/cm^2, and from room temperature to 750 ^{o} C. Generally, bubbles were observed in both the major WC phase as well as the minor Fe phase, with bubbles in WC typically a factor of 2 or so smaller.

Bio-inspired self-healing materials based on ceramic-polyurethane hybrid composites

Researcher : Eleonora D'Elia
Supervisor : Prof Eduardo Saiz
Sponsor : Office of Naval Research (ONR) and DARPA

Biological tissues such as bone and nacre show remarkable properties such as high strength and toughness and the ability to self-repair. To a large degree the unique performance of natural materials is due to the presence of thin, interfacial organic layers. Therefore, mimicking natural hierarchical organic/inorganic structures requires careful engineering of the interfacial adhesion between the components based on a deep understanding of the role of the organic "soft" phase. In this work we test simple-model silica glass/polyborosiloxanes interfaces that exhibit self-healing properties based on the use of interfacial sacrificial hydrogen bonds. The healing process is divided in two steps, one is the reforming of bonds and the other is the spreading of the soft phase to refill the interface. These materials are used to build brick-and-mortar and laminate structures that exhibit toughness higher than the single components. Furthermore, three point bending studies show that these structures are able to heal completely and recover their properties few days after fracture. These results show that the use of a self-healing shear-thickening soft interface is a promising approach to build biomimetic hierarchical structures. The development of this model is instrumental to the realisation of more complex interfaces and structures opening a path to the realisation of structural composites able to self-heal fully and autonomously. The techniques studied will be applied to the creation of new meta-materials able to self-heal, sense and adapt to the environment.



Carbides for Future Fission Environments (CAFFE)

Researcher : Eugenio Zapata-Solvas
Supervisor : Bill Lee
Funder : EPSRC

The aim of this project is to develop new Zr-based carbides, including Zr-based MAX phases, for coating Zr-alloys cladding in fission reactors of future nuclear power plants. He is responsible for the synthesis, sintering by hot press and spark plasma sintering and microstructural characterization by XRD, SEM and TEM of the different Zr-based carbides, which will be supplied to Manchester University and Cambridge University for irradiation and irradiation plus corrosion studies among others. A second stage of the project includes the study of oxidation, corrosion under water pressurized reactor conditions, mechanical and thermal properties of the developed Zr-based carbides. Further areas of interest include the mechanical reinforcement and study of the plasticity of ultra-high temperature ceramics and flash sintering of ceramics.

PhD Projects

Effect of Minor Elements on Calcium Aluminate Cement Refractories

Researcher: Jennifer Alex
Supervisors: Luc Vandeperre, Bill Lee
Sponsor: Kerneos Ltd, France

The role of various oxides on the solid-liquid interactions in calcium aluminate cements (CACs) at high temperatures is being examined using phase and microstructural characterisation (SEM, TEM, XRD and TGA) and Gibbs energy minimisation computational modelling. In light of recent trends in the refractory industry to replace bricks by higher purity CAC-based monolithics for enhanced durability, performance and novel applicability, improved mineralogical control is of growing significance. The effect of minor elements on the development of a microstructure within these systems during processing and application is being examined with emphasis on potential mineralisation effects of liquid formation linked to these elements as well as the grain growth which occurs through dissolution/precipitation reactions. Furthermore, threshold values are being determined above which the presence of minor elements leads to a deterioration of desirable refractory castable properties.

Graphene–based composites for pipeline liners and coatings

Researcher : Alan Leong
Supervisor : Eduardo Saiz and Cecilia Mattevi
Sponsor : PETRONAS, Malaysia

Graphene is a 2D material with unique functional and mechanical properties, from tuneable electrical and optical response to high intrinsic stiffness and strength, chemical versatility, controllable permeability or extremely high specific surface area. It has the potential to revolutionize a wide range of technologies from batteries to composites and membranes. However, to achieve this goal we need to develop ways to integrate graphene into fabrication technologies and to develop approaches to synthesize large quantities of material tailored for specific applications.

Graphene is a very appealing reinforcing phase for polymer pipeline liners/coatings. The addition of graphene can provide controlled permeability, enhanced mechanical properties and even sensing capabilities. However, neither mechanical exfoliation nor chemical vapour deposition are amenable to the large-scale synthesis of graphene needed for this application. We will address this need by using chemically modified graphene (CMG) that can be fabricated in bulk quantities. CMG intrinsic surface area, permeability and mechanical properties are comparable to pristine graphene and combined with its unique chemical versatility it opens exciting possibilities for the development of novel composites.

Oxidation of Carbides Including Carbide Nuclear Fuels

Researcher : Claudia Gasparrini
Supervisors : Bill Lee, Luc Vandeperre
Sponsor : EPSRC as part of DISTINCTIVE (Decommissioning, Immobilisation and Storage SoluTIons for NuClear wasTe InVEntories) consortium.

The UKs inventory of exotic fuels includes some carbides for which no long term waste management decision has been made. The aim of this project is to get knowledge on the oxidation mechanism of carbide fuels, and develop a suitable oxide wasteform. Despite the importance of the understanding of oxidation in uranium carbide (UC) fuels, this material was not readily available for experimental work. To understand the oxidation mechanism of carbide fuels, baseline work has been performed on an inert carbide material, zirconium carbide (ZrC). Oxidation experiments in the range of temperature of 800-1200˚C have been performed on hot pressed specimens of ZrC using a TGA and a chamberlift furnace for the evaluation of the kinetic of the reaction. Investigations on the mechanism of the reaction have been carried out via microstructural characterization via SEM, FIB-SIMS and TEM on samples cross sections on not fully oxidized specimens. *In-situ* experiments have also been performed in a HTESEM (High Temperature Environmental SEM) in collaboration with the ICSM (Institut de Chimie Séparative de Marcoule), France. The monitoring of the *in-situ* reaction coupled with Finite Element modelling enabled the understanding of the formation of the “Maltese cross” shape of the oxide (see figure 1).

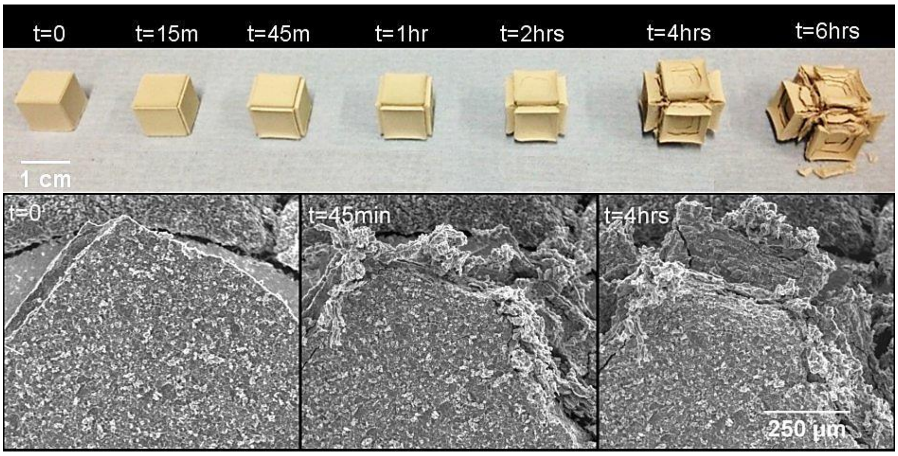


Figure 1. ZrC specimens oxidized in a chamber lift furnace at 800 ˚C followed by a SE sequence of images of a sample oxidized at 800C in a HTESEM: details of the cracks formation and opening of cube edges leading to the Maltese Cross

The preliminary work on ZrC will be used for comparison and understanding of the oxidation mechanism of depleted uranium carbide spent fuel coming from Dounreay facility. Characterization and oxidation studies on UC are performed using NNL (National Nuclear Laboratory) facilities, Preston, UK. Experiments involve characterization of the initial material via XRD and SEM-EDX and oxidation experiments performed in a TGA/MS in the range of temperatures of 700-1200˚C. The oxidation products will be then characterized via XRD, SEM-EDX and via BET. In addition, the understanding of the oxidation mechanism of mixed carbide fuels will be carried out on a surrogate of (U,Pu)C.

Study of Phase Evolution and Properties of a Porcelain Body Fabricated Using Different Rapid Firing Techniques

Researcher : Wirat Lerdprom
Supervisor : Bill Lee and Doni Daniel
Sponsor : Cementhai Ceramics Co, Ltd., Thailand

The project aims to build up an understanding and new knowledge of clay-based ceramic microstructure and properties, i.e. porcelains, which is sintered by different types of rapid firing techniques, in order to find a possibility to reduce the energy consumption of the existing ceramic production process.

Many efforts have successfully utilized the rapid sintering techniques such as spark plasma sintering (SPS), flash sintering (FS) and microwave sintering (MWS) for sintering various kinds of ceramics. However, there is a few of those techniques dealing with clay-based ceramic bodies in order to study microstructure and sintered phase compositions which represents their technological properties.

In this study, a porcelain body is sintered using SPS, FS and MWS with designed experimental parameters. The densification behaviour which is expressed by apparent bulk density, water absorption etc., and the final phase compositions are investigated. Since, the porcelain body is composed mainly of kaolinitic clays, feldspars and quartz; thus the phase evolutions including metakaolin formation, mullite formation, glass phase formations and quartz dissolutions will be discussed. Moreover, comparisons of the products fabricated from those techniques in terms of energy use, financial and other aspects will be determined which is most likely to find industrial application.

In Situ Fracture Tests of Brittle Materials at The Microscale

Researcher : Giorgio Sernicola
Supervisors : Finn Giuliani and Ben T Britton
Sponsor : Element Six

The fracture toughness of ceramics is often dominated by the structure of their grain boundaries. Our ability to improve life of ceramic components depends on our ability to investigate properties of individual grain boundaries. This requires development of new fracture testing methods allowing high spatial resolution and high control over the area to test. Further benefits of these ‘small scale’ approaches will enable testing of specimens for which big volumes are not available (e.g. thin films, coating, or simply samples of dimensions limited by production process).

Recently, several techniques have been developed using small scaled mechanical testing, based within a nanoindenter, changing tip and sample geometries, including: micropillar compression; microcantilever bending; and double-cantilever compression. However, the majority of the published works utilises complex geometries resulting into complex analysis of force distribution and stress intensity factor and rely on load-displacement curves for the identification of crack initiation, with the added complication of friction.

Our approach builds upon the work of Lawn, who showed that a practical test geometry to obtain stable crack growth and calculate the fracture energy G is that of a double-cantilever beam (DCB) under constant wedging displacement. We replicate this configuration in our tests fabricating double-cantilever beams of micrometric dimensions by focused ion beam (FIB) milling and loading them in-situ in an SEM using a nanoindenter with a wedge-shaped tip. This has two benefits: the sample is well aligned for a controlled test; images are recorded during the test for later analysis. This allows us to use beam deflection and crack length rather than critical load to measure fracture toughness. Our tests have proved it is possible to initiate and stably grow a crack in a controlled manner in ceramic materials and our fracture energy results have been validated against prior macro-scale fracture data. This approach is being extended to multi-phase materials with unknown materials properties and extends our arsenal of small-scale characterisation techniques required to generate new processing strategies for the next generation of materials design.

Development of a Novel Wound Management Dressing

Researcher : Annalisa Neri
Supervisor : Eduardo Saiz
Sponsor : Welland Medical Ltd.

Wound management represents a challenging area, both in research and product development and in clinic. An active dressings able to promote healing while preventing infection is necessary in order to address several wound types in particular burns and chronic wounds. The focus of this study lies in the development of a novel collagen-honey dressing for such aim. These two components indeed seems to have promising properties in the wound care field, therefore it is worthy to explore the potentials of their synergetic effects in wound healing. During the first phase of this project the processing optimisation for producing plain and smooth collagen/honey films with different compositions was achieved. Currently their characterisation is taking place. In particular their response in terms of swelling and degradation is being investigated along with their homogeneity, mechanical properties and honey release kinetic. Furthermore viability and proliferation of human skin fibroblasts is being assessed. In the future work further studies regarding enzymatic degradation, denaturation temperature and the possibility to couple collagen with other materials in order to modulate and enhance its properties will be investigated.

Bio-inspired ceramic-based composites

Researcher : Claudio Ferraro
Supervisors : Eduardo Saiz, Julian Jones
Sponsors : Marie Curie ITN –FP7 project

Ceramics exhibit outstanding properties from high chemical and thermal resistance to high hardness and strength that made them suitable for different applications. However ceramic materials are susceptible to brittle fracture, drawback that has partially limited their use. Traditional structural ceramics show low values of fracture toughness and therefore new production methods and strategies are needed to produce structural ceramics with higher fracture resistance. Taking inspiration from nature, and more specifically from the peculiar structure of nacre (mother of pearl), composite materials based on freeze cast ceramics have been studied. The process of freeze casting, which uses ice crystals as template, has been used to obtain fine lamellar porous ceramic scaffolds. In order to mimic the nacre composite structure, where the ceramic part is alternated in a layered structure with a polymeric soft phase, the porosity of the freeze casted scaffolds has been infiltrated with a polymeric (PMMA) or a metallic (Aluminium) second phase. Two different types of ceramics have been considered, alumina (Al2O3) and silicon carbide (SiC). The mechanical properties of these composites have been characterized in terms of flexural strength, fracture toughness and as well in terms of R-curve behavior. Due to the interlayered structure these composites exhibits rising fracture toughness (up to ≈20 MPa·$m^{1/2}$) with the crack propagation. Freeze casting has been used also to produce highly porous SiC lattices (porosity up to 98 vol%) with excellent crushing strength (2.5MPa). These foams have been characterized in terms of mechanical, thermal and electrical resistance.

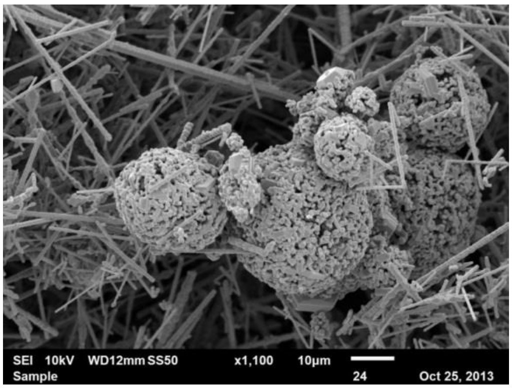


Figure 1 CVD deposition of SiC spheres during the sintering at high temperatures of SiC scaffolds

Calcium phosphate scaffolds with controlled composition and structural properties for biological applications

Researcher : Gil Costa Machado
Supervisor : Eduardo Saiz
Sponsor : Marie Curie ITN –FP7 project

After the initial characterization of the starting powders, the thermal behaviour of the materials with a biphasic composition (from pure Hydroxyapatite to pure β-Tricalcium Phosphate) was assessed and the optimal sintering conditions were established. Furthermore, an innovative emulsion-based soft templating method was used for the purpose of controlling structural features (porosity, pore size and interconnectivity) at the micrometric scale. The biological behaviour of such materials was studied *in vitro* by incubating them with human mesenchymal stem cells in an osteogenic medium. Specific differentiation markers were measured to assess the osteogenic potential of the Calcium phosphate flat substrates with varying roughness profiles. Three-dimensional scaffolds were also produced through robocasting periodical structures with the emulsion based slurries. Printing conditions were optimised for different compositions and their bioactivity was evaluated through standard incubation tests in Simulated Body Fluid.

An important goal of this work was to fabricate three-dimensional scaffolds with these materials - i.e., incorporating the microporosity and roughness that results from the emulsification process – while controlling structural features at a macrometric scale, such as pores with hundreds of micrometers where cells can penetrate and proliferate. For such processing to be possible, it is paramount to understand the rheological behavior of these materials. Fig. 1 shows how the apparent viscosity of the ceramic suspension and emulsion at different steps of the processing route varies with the shear rate. At basic pH (early in the process), the materials are very fluid. However, in order to be used in 3-D shaping techniques such as robocasting, they must simultaneously be fluid enough to flow through a nozzle during extrusion and be stiff enough to keep their shape after that. Therefore, the coagulation step (pH switch) previously mentioned is necessary, as it increases the viscosity of the materials roughly three orders of magnitude, making them suitable for 3D printing.

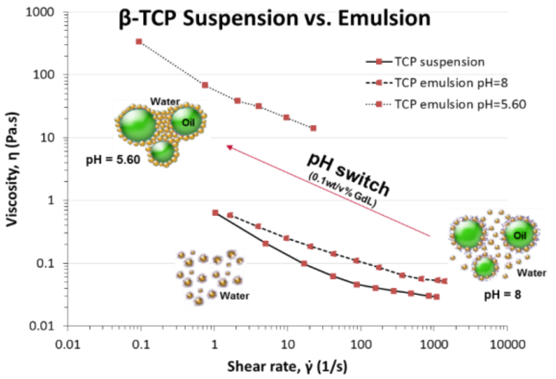


Figure 2 – Apparent viscosity of both initial (TCP suspension) and emulsified (TCP emulsion) ceramic suspensions, and coagulated (TCP emulsion pH=5.60) emulsion after pH switch

At this point, it is possible to make large scaffolds with controlled structures and geometries by extruding these particle stabilized emulsions. Fig. 9 shows a piece prepared through such method, on which it is possible to see the microporosity that results from the oil droplet templating and how it reflects of the final surface roughness of each printed rod. These levels of structural control allow for an extremely versatile method of production of calcium phosphate scaffolds with varying chemical composition.

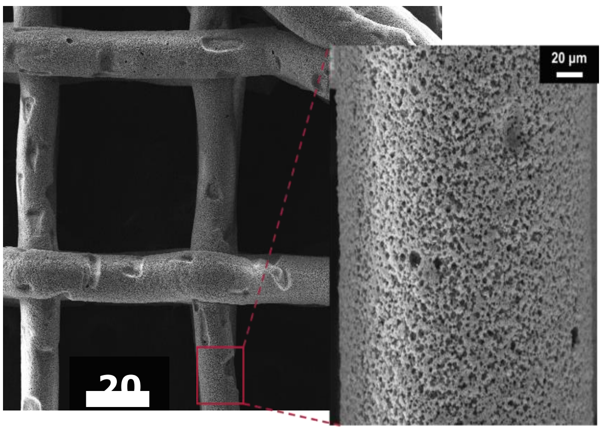


Figure 3 – Robocasted scaffold by extruding particle stabilised emulsion with HA

3D Printing of Ceramic Composites

Name : Ezra Feilden-Irving
Supervisors : Eduardo Saiz, Finn Giuliani, Luc Vandeperre
Sponsor : CASC - Industrial Consortium

3D printing has shown great promise over the last two decades for the production of geometrically complex, monolithic ceramic parts. Despite this, the mechanical properties of printed ceramics are often poor due to defects incurred during printing. We have addressed this issue by tailoring the printing paste and parameters, and have demonstrated dense printed material with properties comparable to slip-cast parts.

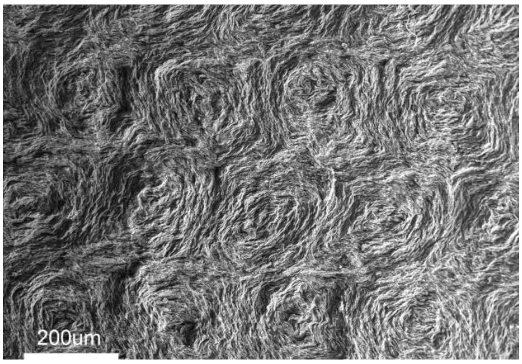


Fig.1 – Fracture surface of a platelet-epoxy composite where the printing process has been used to create concentric platelet alignment mimicking the structure of bone

Attention has now turned to the potential for 3d printing to be used to create ceramic composites with novel, complex microstructures, controlled by the patterning process itself. This is achieved by using the extrusion forces during printing to align anisotropic particles such as short fibers or platelets, followed by infiltration by the matrix phase. Fig. 1 is an example of one such microstructure. This level of meso-scale control allows for the exploitation of toughening mechanisms similar to those seen in natural materials. The metal and polymer matrix composites exhibit high strength and toughness, and can be printed into virtually any shape.

Potential Wasteforms for Immobilisation of Advanced Reprocessing Wastes

Researcher : Yun-Hao Hsieh
Supervisor : Bill Lee
Sponsor : The UNSW Tyree Scholarship

A number of processes are being developed to improve current reprocessing technology (plutonium uranium redox extraction), PUREX, by further separating the remaining waste. A promising method is the so-called Group Actinide Extraction (GANEX) process, which primarily recycles major and minor actinides together and is thus more proliferation resistant than other strategies (Pu is never found alone at a weapon-grade level).

A novel development of GANEX, called EURO-GANEX, has been designed and tested at NNL (National Nuclear Laboratory, UK) and JRC-ITU (Joint Research Centre- Institute for Transuranium Elements, Germany). Both EURO-GANEX tests at both institutions got very high recoveries of transuranium product (e.g. higher than 99.8% of recoveries in actinides and minor actinides at JRC-ITU hot cell test). The remaining waste stream is therefore likely to be just fission products (FPs). This modification requires new immobilization matrices to be developed that are tailored to accommodating actinide-free waste streams.

A potential wasteform for immobilizing HLW from EURO-GANEX is Synroc (Synthetic rock). Synroc is a family of advanced crystalline ceramics comprised mainly of 4 titanate phases: hollandite, perovskite, zirconolite and rutile. Each phase incorporates a different set of radioactive waste elements, therefore it is possible to tailor the matrices of Synroc to particular waste streams by adjusting the ratio of the different titanate components.

Since the zirconolite phase in Synroc mainly hosts actinides, the proportion of this phase present can be greatly reduced in waste forms designed for immobilizing HLW from EURO-GANEX reprocessing. This can be achieved by reducing CaO and removing ZrO_2 from the blend of starting powders. This will result in an increase in the level of other phases present and therefore increase the waste loading of the modified wasteform. Such modified Synroc samples have been successfully produced, their phase composition characterized, and their microstructures determined. Samples containing simulated EURO-GANEX HLW elements were also produced. Waste simulants were found to be incorporated into the Synroc structure, as designed.

Multi-scale predictive modelling of ultra-high temperature structural ceramics

Researcher : Michele Pettinà (Mechanical Engineering)
Supervisors : Kamran Nikbin (Mechanical Engineering) Luc Vandeperre (Materials Department)
Sponsor : Defence Science and Technology Laboratory (Dstl)

The project, supervised by Prof. Kamran Nikbin and Prof. Luc Vandeperre and funded by the UK's Defence Science and Technology Laboratory (Dstl), is about ultra-high temperature ceramics (UHTCs). The primary aim of the project is to develop computational methods to assess damage and failure in UHTCs and aid in the design of laboratory tests, thus reducing time and costs associated with experiments. UHTCs are considered attractive candidates for nose cones and sharp leading edges of next generation hypersonic re-entry vehicles thanks to their high melting point, good stability at high temperature and high thermal and electrical conductivity. However, UHTC components may undergo creep and surface oxidation while operating at high temperature in aggressive environment, as well as fail prematurely due to thermal shock.

A continuum damage mechanics-based model running as a custom user subroutine in the finite element (FE) software Abaqus has been proposed to account for damage due to creep and time-dependent material oxidation. Initially, the model has been applied to a representative three point bend geometry to illustrate the methodology proposed to predict creep damage. Abaqus user subroutine was later extended to include a simple novel approach to estimate oxidation damage. Additionally, a microstructural model has been incorporated in the FE code to be able to treat grains and grain boundaries separately in the analysis. By assigning different material properties and oxidation rates to grains and grain boundaries, enhanced diffusion to grain boundaries observed experimentally on ZrN samples oxidized in the temperature range 900-1100°C has been modelled successfully. More recently, the same approach has been used to predict the heat flow, temperature distribution, phase transition and stress distribution during high heat flux laser tests of HfB_2 samples and compared with experimental results. Results have been presented at several international conferences, such as ICACC'13, CERMODEL 2013 and 2015, CIMTEC 2014 and at the UHTC III meeting in Australia.

Silicon doped boron carbide as a lightweight impact resistant material

Researcher: Cyril Besnard
Supervisors: Luc Vandeperre and Finn Giuliani
Sponsor: DSTL

The project is supported by the Defence Science and Technology Laboratory of the UK. The aim is to develop novel ceramics for use in armour. Lightweight impact resistance ceramics are still under development. B4C is attractive and has already used for this application for many decades. However a catastrophic failure occurs in B4C at the high pressures achieved during high velocity impacts, which is due to collapse of a weak polytype within the structure. Previous research has suggested that doping with silicon can eliminate this polytype and therefore improve the high velocity impact performance of B4C. Therefore the aim of this project is to produce meaningful quantities of Si doped B4C which can be used for high speed impact testing. This project will be in collaboration with the shock physics group at Imperial College.

Optimising Hard Coating Design by Experimental Investigation and Microstructural Modelling.

Researcher : Tommaso Giovannini
Supervisor : Finn Giuliani, Daniel Balint
Sponsor : SECO Tools, Sweden

Metal cutting tools are subjected to extremely harsh operational conditions in the form of high temperatures and mechanical loads which can lead to severe wear of the tool. Because of these conditions, thin ceramic coating layers are applied to metal cutting tool surfaces using chemical and physical deposition methods. These coatings are used to improve wear resistance, chemical inertness and the mechanical properties of the tool as a whole. The coatings which are being investigated on this project are usually composed of two or more layers which are made of titanium nitrides and carbides (TiN & TiCN) as well as textured alumina ((100), (012), (001) Al2O3).

The majority of the worked aimed at improving coating design thus far has mainly focused on a trial and error approach and lacks thorough understanding of the physical mechanisms which govern wear at these reduced length scales. The aim of the project is to gain this understanding using both an experimental and computational framework. The two main aspects which are under investigation are plastic behaviour of the coatings at increasing temperatures and variations in the quality of adhesion between the coatings and the substrates according to different processing parameters.

The investigation is composed of both experimental and computational frameworks. The experimental work is based on *in situ* micro-mechanical testing within a scanning electron microscope (SEM). Coating micropillars are previously manufactured, using FIB, then placed within a purposely built loading stage which is placed inside the SEM chamber. The micropillars are then compressed and fractured at increasing values of temperature to simulate the conditions present at the cutting tool interface. These tests provide valuable insights with regards to the coating properties and allow for the extraction of parameters which can be used as inputs in the discrete dislocation (DD) modelling framework.

The modelling framework is based on planar discrete dislocation dynamics and serves as a complement to the experimental work to shed light on the microstructural mechanisms which lead to the mechanical response of the coatings under different loading regimes. This is done by adjusting the boundary conditions to simulate the experiments which are carried out as well as a real cutting operation.

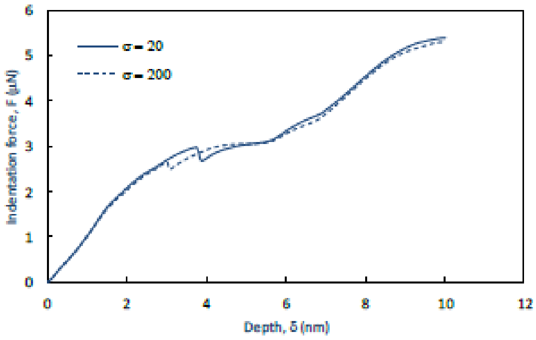

Roland

Capabilities and facilities

The purchasing and installation of large items of equipment by the Centre from the original CASC project, to improve UK capability in fabrication and modelling of structural ceramics, is now complete but we continue to improve our experimental capability in this area using funds from other sources. All equipment is available to the UK ceramics community – please contact Amutha Devaraj (adevaraj@imperial.ac.uk, 020 7594 1170) or Garry Stakalls (g.stakalls@imperial.ac.uk, 020 7594 6770) if you wish to use any of these facilities.

Nanoindenter

The high temperature nanoindenter manufactured by Micro Materials is located in the Structural Ceramics laboratory on the lower ground floor of RSM, to make use of the better control of air temperature and reduced vibration level. As well as being fully instrumented, the nanoindenter operates at temperatures up to 750 °C. Usage of the nanoindenter is high, and results obtained (Section 7) have been reported at international meetings including the Third International Workshop on Mechanical Behaviour of Systems at Small Length Scales, Kerela, India , Fall MRS conference, Boston, American ceramics society meeting, Daytona beach, ICMCTF San Diego. There is also an ongoing projects with SECO Tools AB and Element 6 (total value ~£65K).



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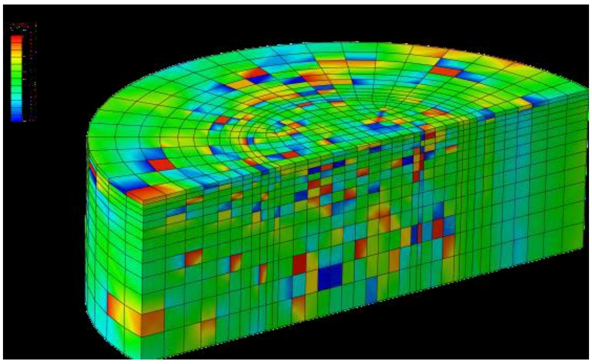


CASC's multiprocessor server is being used to model the response of MgO under nanoindentation. Three dimensional crystal plasticity simulations are being carried out using parallel processing on the CASC cluster. The relation between primary and secondary slip systems activation and hysteresis, and the softening observed in the indentation force displacement response, have been simulated. A normal random distribution of the critical resolved shear stress results in a lower drop in the indentation force. This is being used to study the relation between the change in the slope of the loading curve (corresponding to the activation of the secondary slip systems) and the spacing of hysteresis loops observed in the experimental data.

Freeze dryer

Freeze-drying is a drying process, where the solvent, normally water, is eliminated from the sample via direct sublimation from solid to gas phase. This is a useful way to eliminate the solvents by keeping the material structures intact for further processing like sintering. We currently use this process for drying freeze-cast materials like alumina, zirconia, zeolites and graphene oxide.

Thermodynamic software

FactSage version 6.1, together with three substance databases, has been purchased from GTT Technologies. A multi-user license for phase equilibria software has also been purchased from the American Ceramic Society. This thermodynamic calculation software is available over the network to anyone in the CASC offices and has been applied to range of projects including Si-stabilised B_4C and high temperature annealing of TiAlN, thermal treatments of high alumina castable refractories, producing composites of B4C and SiC.

Thermal analysis

A suite of high-temperature thermal analysis equipment from Netzsch has been installed in the Department of Materials, in a basement room that was converted specifically for this use. The equipment comprises:

- Simultaneous TG-DTA to 2000 °C
- Laserflash (thermal diffusivity) to 2000 °C
- Dilatometer (thermal expansivity) to 2400 °C


Crucibles of the high temperature combined TGA DTA

Netzsch have provided multiple training sessions, and all three items of equipment now run at temperatures up to 2000 °C. The facility is heavily used and starting to attract external users.
The **dilatometer** has two set-ups: an alumina tube and pushrod for measurements up to 1600 °C and a graphite set-up for measurements up to 2400 °C. In-house developments in the past year have made it possible to use the dilatometer to measure hardness too and initial tests have been run to use it for creep measurements. Examples of CASC projects using the dilatometer are the measurement of the thermal expansion of refractory materials for estimating the risk of thermal shock damage, the characterisation of a wide range of ultra-high temperature ceramics, the study of mullite sintering and the analysis of residual stresses in mullite zirconia composites, hardness measurements of ZrB_2 and Al_2O_3 and the analysis of cracking due to shrinkage in geopolymers and sintering of silicon carbide-boron carbide composites. Measurements for industrial partners such as Rolls Royce have also been carried out.
The **combined TGA-DTA** has been used to quantify mass loss during drying of geopolymers, analyse the decomposition of magnesium phosphate and magnesium silicate cements for nuclear waste treatment, the study of silicon carbide or mullite sintering, the analysis of UHTC oxidation, the determination of carbon yield from various ceramic additives and the characterisation of raw materials in general. Usage for third parties included work with Professor Jon Binner, Loughborough University and Dr Bai Cui at the University of Illinois, as well as characterisation of derivative products from commercial paper mills and work with Morgan Technical Ceramics.
The equipment for measuring **thermal diffusivity via laser flash** has been used extensively to characterise a wide range of ultra-high temperature ceramics and carbon-ceramic composites as well as in collaborations with Rolls- Royce, Morgan Technical Ceramics and Professor Reece at Queen Mary College (thermo-electric materials)

Induction-heated thermo-mechanical test rig

Thermo-mechanical testing

The high-temperature mechanical testing equipment from Instron is located in the Mechanical Engineering Department. One frame incorporates a vacuum system and a furnace from Materials Research Furnaces with a maximum temperature of 2000 °C. The equipment is used in work with diverse industrial partners such as Seco Tools AB, Sweden. The second frame has induction heating up to 1200 °C. In the last years, it has been used for a range of projects including measuring the properties of commercial cutting tools near the service temperature, studying mullite creep, and measuring high temperature strength of UHTC's and commercial refractories .

Vacuum hot press

The vacuum hot press from FCT Systems is now in fully operational. The press will operate at temperatures up to 2400 °C for sintering and 2100 °C for hot pressing with a maximum force of 250 KN at atmospheric pressure or under vacuum. We can use dies with diameters as large as 8 cm allowing the fabrication of large samples. Use for CASC projects include the preparation of a wide range of materials such as silicon carbide, boron carbide and composites of silicon carbide and boron carbide, silicon carbide – aluminium nitride alloys, zirconium carbide, tantalum and hafnium carbide, joining of UHTC's, glass ceramic-SiC composites, ultra-light SiC structures and mullite. The unique high-temperature capability has enabled the fabrication of a solid solution phase of HfC and TaC, which lead to a best poster prize at the ECI conference on ultra-high temperature ceramics at Hernstein, Austria. Runs for other university groups include tests on forging of functional ceramics for Prof Alford, Imperial College London, and treatment of UHTC precursors for Prof Binner, Loughborough University.

Vacuum hot press

Vacuum furnace

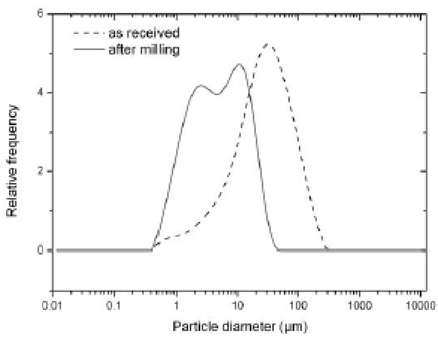
The vacuum furnace from Thermal Technology is now fully operational. The furnace will heat a volume 5 cm diameter and 15 cm tall to temperatures up to 2500 °C under vacuum or a mixture of gasses. Opposed viewing ports will allow observation of the sample during heating, and a sample elevator and cooling chamber will allow exchange of samples whilst the furnace is at temperature. The equipment has been used in the sintering of ceramics and metal-ceramic composites as well as the analysis of glass and metal wetting on ceramic substrates.

Wet grinding mills

We have purchased and installed a wet grinding mill capable of low-amplitude grinding of up to 5kg of ceramics in five different chambers and a ball rolling mill for homogenisation of suspensions and breaking up of agglomerates before processing.

Particle Size Analyser

The Department has provided funds to acquire a laser particle size analyser. The equipment uses scattering of light by particles in dilute solutions to determine the size distribution and has the ability to measure particles with diameters ranging from 10^{-2} to 10^{4} µm without changing the optics.



Examples of powder particles = distributions

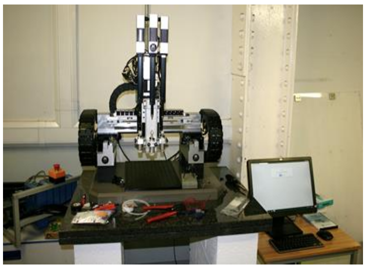
High Temperature elastic properties by impulse excitation

Early May 2013 saw the installation of a new piece of equipment for determining the Young and shear modulus and Poisson ratio of materials. The measurement principle is based on the relationship between shape, density and stiffness and the natural vibration frequencies of a sample. For example, for determination of the Young modulus, typically a bending vibration mode is excited by hitting a sample supported on the nodes of the vibration with a small projectile in the centre. The resulting vibration is picked up with a microphone and analysis of this signal using the Fourier transformation yields the frequency of the vibration. The software also analyses the decay in amplitude of the vibration with time to determine a value for the damping of the vibration. The model installed at CASC comes with a furnace capable of operating to 1750 °C in air or inert atmosphere and hardware and software enabling fully automated excitation and measurement, making it possible to investigate the variation of the elastic properties with temperature and to characterise transitions in the materials behaviour from the changes in internal damping of the vibration signal. Some examples of phenomena giving rise to damping are the glass transition temperature, the hopping of oxygen vacancies bound to dopants in response to stress at low temperature in doped zirconia, and the softening of grain boundary glassy phases in sintered silicon nitride.

Figure Set-up for room temperature measurements.

3D Printer

A robotic assisted deposition system from 3D Inks (USA) has been installed recently in the CASC. The system can print 3D structures using continuous extrusion, with submicron reach submicron-printing precision. The printer allows the combination of three different inks to fabricate multiphase structures.



Optical Microscope Axio Scope A1

Optical microscope with reflected and transmitted light, bright and dark field, DIC, camera and software for image acquisition and analysis is installed recently. The microscope has a modular design that will facilitate the installation of different set-ups to do in-situ experiments, for example, mechanical testing or freezing of colloids.

Rapid prototype (CNC) milling machine

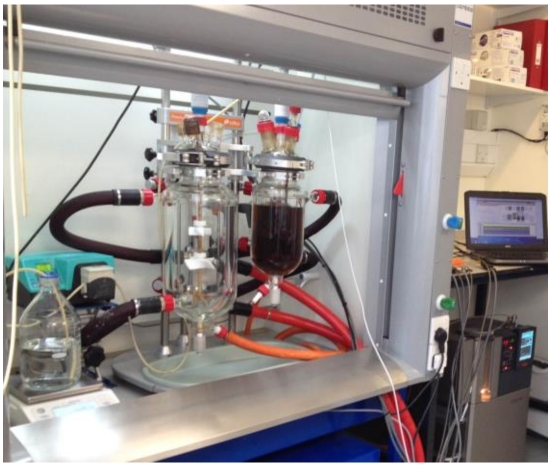
Rapid Prototyping is dramatically transforming the design and manufacturing processes and this milling machine has answered the call for a cost-effective, high precision and compact solution. Used to create realistic models, functional prototypes and moulds and is compatible with a wide range of materials. It produces highly accurate parts including those for complex snap-fits from an extensive range of non-proprietary materials including Acetal, ABS, chemical woods, acrylic, plaster, nylon, styrene and many medical grade materials including PEEK.

It offers a number of significant advantages over additive rapid prototyping (ARP) or "3D printing" systems, making a combination of the two technologies the perfect prototyping solution

Elemental Mass Gas Analyser

An Oxygen-nitrogen-hydrogen Elemental Mass Gas Analyser (Horiba, EMGA – 830 series) has been installed in the CASC in 2015. This also includes Carbon-sulphur Elemental Mass Induction Analyser (EMIA series) and Glow Discharge-Optical Emission Spectroscopy (GD-OES) setup.

Graphene reactor



A one of a kind modular system for the large-scale synthesis of chemically modified graphene based on chemical graphite exfoliation has been installed. This system is flexible and allows “on demand” fabrication of materials with tailored properties. The rig consisted of two jacketed glass reactors of up to 5L mounted on a bench standing framework (*Radleys, Essex, UK)*. Overhead stirrers (*Heidolph*) with PTFE propeller stirring paddles placed at different heights ensured vigorous mixing in the reactors. Oil in jackets was connected to a *Huber Unistat* recirculating chiller. Manipulation of liquids (e.g. addition of concentrated acids or transfer of slurry between vessels) was carried out using a software-controlled peristaltic pump with acid resistant tubing (Marprene). *AVA* software allowed online control of temperature in jacket oil or reacting mixture, mass addition and stirring. The component parts of the system are a computer controlled reactor system with two chambers to perform the chemical exfoliation of graphite at controlled temperature, stirring speed etc. and a purification system based on centrifugation at controlled temperature. This unique modular approach allows us the flexibility to synthesize materials on demand for the different applications.

Other equipment

Other equipment like a new polishing machine and glove box were installed.

An additional set up has been installed with the DTA/TG analyser to measure the specific heat capacity. This is a high accuracy rhodium furnace and the temperature can go up to 1450^{o}C.

CASC Research Portfolio

Funded proposals (Jan – Dec: 2015)

BIOBONE: Bioceramics for bone repair
Funder: Commission of the European Communities
Project start date: 01 March 2012
Budget (£): 2,952,816

Development of a novel wound management dressing
Funder: Welland Medical Ltd
Project start date: 01 April 2014
Budget (£): 41,766

Engineering with graphene for multi-functional coatings and fibre-composites
Funder: Engineering and Physical
Science Research Council (EPSRC)
Project start date: 01 February 2013
Budget (£): 286,893

Graphene Enhancement of the Photocatalytic Activity of Semiconductors (GRAPES),
Funder: Commission of the European Communities
Project start date: 01 December 2013
Budget (£): 164,153

Graphene-based composites for pipeline liners
Funder: Petronas Research Sdn Bhd
Project start date: 01 June 2014
Budget (£): 479,863

Graphene three-dimensional networks
Funder: Engineering and Physical Science Research Council (EPSRC)
Project start date: 01 February 2013
Budget (£): 1,257,620

RESLAG: Turning waste from steel industry into valuable low cost feedstock for energy intensive industry
Funder: European Commission
Project start date: 01 September 2015
Budget (€):8,092,712

Bio-inspired self-healing materials based on ceramic-polyurethane hybrid composites
Funder: DARPA-USA
Project Start date: 31 July 2015
Budget ($):348,069

Carbides for Future Fission Environments (CAFFE)
Funder: EPSRC
Project start date: 2015
Budget (£):£554,707 to Imperial

High Emissivity Coatings for Furnace Linings
Funder: SCG Thailand
Project start date: 2015
Budget (£):497,381

Advanced Waste Management Strategies for High Dose Spent Absorbents
Funder: EPSRC
Project start date: 2015
Budget (£):250K to Imperial

Visiting Fellow Michel Barsoum
Funder: Leverhulme Trust
Project start date: 2015
Budget (£):15,800

Ceramic materials and shaping technologies for short life propulsion systems, Phase II,
Funder: Mictroturbo
Project start date: 2015
Budget (£): 177k

Centre for Advanced Interfacial Materials Science (AIMS)
Funder: Shell Global Solutions International BV
Budget (£) : 1,919,535

Publications: Journal Papers (Jan – Dec: 2015)

1. *Effect of microstructure and grain boundary chemistry on slow crack growth in silicon carbide at ambient conditions,* Al Nasiri, N., N. Ni, E. Saiz, J. Chevalier, F. Giuliani, and L.J. Vandeperre, Journal of the European Ceramic Society, **35**(8): p. 2253-2260, 2015.
2. *Effect of microstructure and slow crack growth on lifetime prediction of monolithic silicon carbide,* Materials Science and Engineering a-Structural Materials Properties Microstructure and Processing, Al Nasiri, N., E. Saiz, F. Giuliani, and L.J. Vandeperre, **627**: p. 290-295, 2015.
3. *ATR-FTIR measurements of albumin and fibrinogen adsorption: Inert versus calcium phosphate ceramics,* Boix, M., S. Eslava, G.C. Machado, E. Gosselin, N. Ni, E. Saiz, and J. De Coninck, Journal of Biomedical Materials Research Part A, **103**(11): p. 3493-3502, 2015.
4. *Self-Healing Graphene-Based Composites with Sensing Capabilities.* Advanced Materials, D'Elia, E., S. Barg, N. Ni, V.G. Rocha, and E. Saiz,. **27**(32): p. 4788-4794, 2015.
5. *A silicon carbide lollipop.* Materials Today, Ferraro, C., E. Garcia-Tunon, and E. Saiz, **18**(2): p. 117-118, 2015.
6. *Printing in Three Dimensions with Graphene.* Advanced Materials, Garcia-Tunon, E., S. Barg, J. Franco, R. Bell, S. Eslava, E. D'Elia, R.C. Maher, F. Guitian, and E. Saiz, **27**(10): p. 1688, 2015.
7. *Joule Heating Characteristics of Emulsion-Templated Graphene Aerogels,* Menzel, R., S. Barg, M. Miranda, D.B. Anthony, S.M. Bawaked, M. Mokhtar, S.A. Al-Thabaiti, S.N. Basahel, E. Saiz and M.S.P. Shaffer, Advanced Functional Materials, **25**(1): p. 28-35, 2015.
8. *Understanding Mechanical Response of Elastomeric Graphene Networks,* Ni, N., S. Barg, E. Garcia-Tunon, F.M. Perez, M. Miranda, C. Lu, C. Mattevi and E. Saiz, Scientific Reports, **5,** 2015.
9. *An Investigation of the Mineral in Ductile and Brittle Cortical Mouse Bone,* Rodriguez-Florez, N., E. Garcia-Tunon, Q. Mukadam, E. Saiz, K.J. Oldknow, C. Farquharson, J.L. Millan, A. Boyde, and S.J. Shefelbine, Journal of Bone and Mineral Research, **30**(5): p. 786-795, 2015.

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11. *Bioinspired structural materials,* Wegst, U.G.K., H. Bai, E. Saiz, A.P. Tomsia, and R.O. Ritchie, Nature Materials, **14**(1): p. 23-36, 2015.
12. *Ultralight, Strong, Three-Dimensional SiC Structures,* Chabi, D., V. G. Rocha, E. Garcia-Tunon, C. Ferraro, E. Saiz, Y. Xia, and Y. Zhu, ACS Nano, 2015. **DOI: 10.1021/acsnano.5b05533**.
13. *Microstructural Evolution of HfB_2–based Ceramics during Oxidation at 1600^oC to 2000^oC,* DD Jayaseelan, E Zapata-Solvas, CM Carney, A Katz, P Brown and WE Lee, Adv. in Applied Ceramics. **114** [5] 277-295, 2015.
14. *Development of Multi-Layered Thermal Protection System (TPS) For Aerospace Applications,* DD Jayaseelan, X Yanda, L Vandeperre, P Brown and WE Lee, Composites, Part B **[79]** 392-405, 2015.
15. *Synthesis of Biopolymer-Derived Zirconium Carbide Powder By Facile One-Pot Reaction*, N Patra, DD Jayaseelan and WE Lee J. Am. Ceram. Soc., **98** [1] 71-77, 2015.
16. *Synthesis of ZrB2/SiC composite powders by a single-step solution process from an organic-inorganic hybrid precursor,* N Patra, DD Jayaseelan and WE Lee, Adv. Applied Ceramics (2015).
17. *Structural and Compositional Analyses of Oxidised Layers of ZrB_2-based ceramics*, DD Jayaseelan, E Zapata-Solvas, R Chater and WE Lee, J. Eur. Ceram. Soc., **35** [15] 4059–4071, 2015.
18. *On the Fabrication of ZrCxNy from ZrO2 via Two-step Carbothermic Reduction-Nitridation*, R Harrison, O Rapaud, A Maitre and WE Lee, J. Euro. Ceram. Soc. **35** 1413-21, 2015.
19. *Second Phase-Induced Degradation of Fused MgO-Partially Stabilised Zirconia Aggregates*, A Quadling, L Vandeperre, M Parkes and WE Lee, J. Am. Ceram. Soc. **98** [4] 1364-71, 2015.
20. *Crystal Structures of Al-Nd Co-doped Zirconolite Derived from Glass Matrix and Powder Sintering*, C-Z Liao, K Shih and WE Lee, Inorganic Chemistry, **54** 7353-61, 2015.
21. *Mechanism and Kinetics of Oxidation of ZrN Ceramics*, R Harrison and WE Lee, J. Am. Ceram. Soc. **98** [7] 2205-2213, 2015.
22. *Low Temperature Solution Synthesis of Nanosized Hafnium Carbide Using Pectin,* N Patra, N Al Nasiri, DD Jayaseelan and WE Lee, Ceramics International, **42** 1959-1963, 2016.
23. *Creep of mullite zirconia composites*, D. Glymond, F. Giuliani, L.J. Vandeperre, Journal of European Ceramic Society, **35**[13], 3607-3611, 2015.
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26. *Second Phase-Induced Degradation of Fused MgO Partially Stabilized Zirconia Aggregates,* A Quadling, L Vandeperre, M Parkes, WE Lee, Journal of the American Ceramic Society 98[4], 1364–1371, 2015.
27. *In situ micropillar deformation of hydrides in Zircaloy-4*, H.E. Weekes, V.A. Vorontsov, I.P.Dolbnya, J.D. Plummer, F. Giuliani, et al., Acta Materialia, **92**, 81-96, 2015.
28. *Fracture Toughness of Porous Material of LSCF in Bulk and Film Forms,* Z Chen, X Wang, F Giuliani and A Atkinson, Journal of the American Ceramic Society, **98** (7) 2183–2190, 2015
29. *Microstructural characteristics and elastic modulus of porous solids,* Z Chen, X Wang, F Giuliani, A Atkinson, Acta Materialia, **89,** 268-277, 2015
30. *Analyses of Microstructural and Elastic Properties of Porous Electrodes Based on Focused Ion Beam Tomography,* Z Chen, X Wang, F Giuliani and A Atkinson, Journal of Power Sources **273**, 486-494, 2015.

31. *Stabilization of Boron Carbide via Silicon doping,* J Proctor, V Bhakhri, R Hao, T Prior, T Scheler, E Gregoryanz, M Chhowalla, and F Giuliani, F. Journal of Physics: Condensed Matter, **27,** 2015

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33. *Processing and Properties of ZrC, ZrN and ZrCN Ceramics: A Review,* RW Harrison and WE Lee, Adv. Applied Ceramics, (2015) in print.
34. *Effect of Oxidation on Mechanical Properties of Ultra-high Temperature Ceramics*, E Zapata-Solvas, DD Jayaseelan, PM Brown and WE Lee, Accepted, J. Euro. Ceram. Soc., (2014).
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36. *Tungsten Carbide is More Oxidation Resistant than Tungsten When Processed to Full Density*, SA Humphrey-Baker and WE Lee, Scripta Mat. (2015).
37. AeroSiC: Light and Strong SiC Micro-lattices, C. Ferraro, E. Garcia-Tuñon, V. G. Rocha, S. Barg, M. D. Fariñas, T. E. Gomez Alvarez-Arenas, G. Sernicola, F. Giuliani and E. Saiz. Accepted in Advanced Functional Materials

Outreach

Newsletters

The CASC newsletter per annum, together with the annual report, provides news and contact information for visitors to the Centre and for dissemination at meetings and international visits. Two newsletters were circulated in March and November 2015, covering additional CASC research and equipment, visitors to the Centre and the second summer school on ceramics.
(http://www.imperial.ac.uk/media/imperial-college/research-centres-and-groups/centre-for-advanced-structural-ceramics/CASC-2015.pdf
http://www.imperial.ac.uk/media/imperial-college/research-centres-and-groups/centre-for-advanced-structural-ceramics/Newsletter-8-CASC.pdf)

Website

The website (www.imperial.ac.uk/casc) contains details of CASC staff, visitors, equipment and activities. Meetings organised by CASC, as well as future UK and international ceramic-related meetings are advertised on this website. The previous annual reports and other publicity material can be downloaded from this website. More information about CASC staff, and their research activities and presentations, is being added to the website.

Richard Brook Prize

In 2010, CASC set up the Professor Sir Richard Brook Prize for the best ceramics PhD thesis in the UK with sponsorship from Morgan Advanced Materials. This prize aims to increase the sense of community amongst PhD students researching ceramics in the UK and to mirror the IOMMM's AT Green Award that is available to undergraduates. The 2014 prize was won by Dr James Thomas Bennett's research on *Development of bismuth ferrite derived piezoelectric ceramics for high temperature applications* was supervised by Professor Andrew Bells at University of Leeds. The award in general covers a certificate, plaque and £1000 cheque.

CASC Industry Day

Fifth CASC industry day was held on 23rd January 2015 at Imperial College with attendees from industry and university (Morgan Advanced Materials, Rolls-Royce, ADML, CoorsTek, John Crane, Element Six, Kerneos, Queen Mary University, MoD). The CASC industry day was followed with the Steering Group meeting in the afternoon. The aim is to continue CASC activities and strengthen our relationship to industry.

CASC Summer School on Ceramics

The 6th edition of the **Ceramic Summer School** was held on 16, 17 and 18 September 2015 at Imperial College London. The course was attended by a mix of people from Morgan Advanced Materials, Saint Gobain, the European Space Agency, a range of UK universities and an independent consultant.

The next edition will probably be held 14th - 16th September 2016, but the exact date and programme will be published early in 2016 after input from our industrial consortium.

CASC seminars

The following seminars were arranged in CASC during 2014

27 March 2015	Ultra-fast and energy-efficient sintering of ceramics by electric current concentration Dr E. Zapata-Solvas Materials Science Institute of Seville, CSIC-University of Seville, Seville, Spain
20 April 2015	Thermochemical Stability of Lanthanum Orthoferrite, Oxygen Ion Conducting Membranes Prof Darryl P. Butt Dept. of Materials Science and Engineering, Boise State University, Boise, ID USA
29 May 2015	Reactive Hot Pressing of Zirconium Diboride: Densification Behavior and Thermal Properties Prof W.G. Fahrenholtz Missouri University of Science and Technology, Rolla, USA
October – November 2015	Five Leverhulme Lecture Series and One special lecture - Physical Properties of the MAX Phases - Mechanical Properties of the MAX Phases I: Ambient Temperatures - Mechanical Properties of the MAX Phases II: High Temperatures - Thermal Properties and Oxidation Resistance of the MAX Phases - MAX Phases for the Nuclear Industry: A Preliminary Assessment - A Possible, Partial Solution to the Mystery of the Great Pyramids of Egypt Prof. Michel W. Barsoum, Distinguished Professor in the Department of Materials Science and Engineering at Drexel University during his sabbatical visit supported by Leverhulme grant.