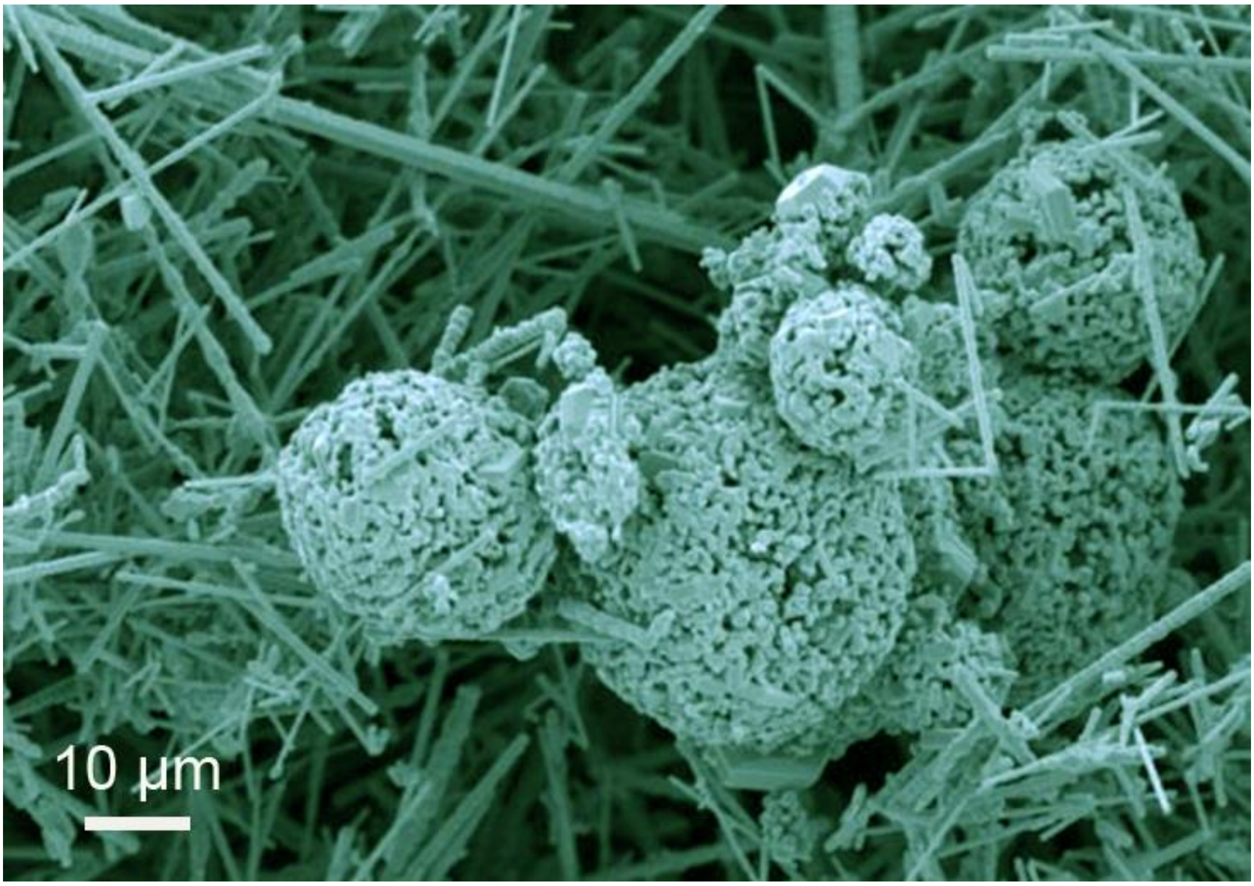
Centre for Advanced Structural Ceramics
Annual Report – 2014



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


Desk 17
Chin Neng Phuah
Desk 18
John O'Neill


I ♥ MATERIALS

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 and PhD students working on projects related to structural ceramics.

Industrial Consortium Group (ICG)

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

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. Our plan involves 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 included. Access 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.
- 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 four members signed up at Sapphire level (Morgan Advanced Materials, DSTL Rolls-Royce and Kerneos) 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

	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

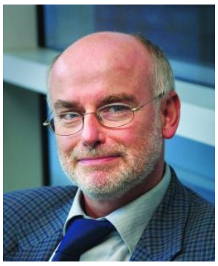
People

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 Niranjan Patra	Research Associate
Dr Laura Larrimbe	Research Associate
Dr Ayan Bhowmik	Research Associate
Dr Nasrin Al Nasiri	Research Associate
Dr Vatish Patel	Research Associate
Dr Sum Wei Siang	Research Associate

PhD students

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

Academic Visitors

Akifumi Niwa	Asahi Glass Co.
Lidia Maria Goyos Ball	CINN University, Oviedo, Spain
Carolina Abajo Clemente	Universidad Carlos III, Madrid, Spain
Dr. Carolina Clausell Teroll	Universitat Jaume I, Castellon, Spain
Masaki Tsutsani	Nagoya Institute of Technology, Japan
Rachid Msaoubi	Seco Tools, Sweden
Dr. Arash Moavenian	Welland Medical
Yulei Bai	Harbin Institute of Technology, China

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)
John Mitchell	Nextec Ltd.
Daniel Glymond	PDRA CASC (Luc Vandeperre)
Rui Hao	PDRA, University of Illinois (in process)
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	Academic staff, Nebraska-Licoln University, USA
Naeem Ur-rehman	PDRA KAUST and now Baker Hughes, SA

Research

PDRA Projects

Fabrication of ternary carbides in Hf-Al-C system

Researcher : Doni J. Daniel
Supervisor : Bill Lee
Sponsors : EPSRC XMat programme grant

A recent progress in theoretical prediction, preparation and characterization of layered ternary transition metal carbides reveals that it is difficult to synthesize single phase ternary carbides in Zr-Al-C and Hf-Al-C systems.[1] Recently, a new family of layered ternary and quaternary compounds with the general formula of (TC)nAl_3C_2 and $(TC)_n[Al(Si)]_4C_3$ (where T = Zr or Hf, n = 1, 2, 3. . .) was developed in Zr-Al(Si)-C and Hf-Al(Si)-C systems.[2-6] The high degree of stiffness retained at elevated temperatures provide Zr-Al(Si)-C compounds with promising applications in high temperature and ultra-high temperature environments.[6] Michalenko et al.[7] and Nowotny et al.[8] have investigated complex carbides in the ternary Hf–Al–C system; two ternary carbides ($Hf_3Al_3C_5$ and $Hf_2Al_3C_4$) were discovered and determined to have hexagonal symmetry with the space group $P6_3/mmc$. Their crystal structures can be described as Hf–C slabs in an NaCl-type structure intercalated by Al_3C_2 blocks, which is similar to the structure of the Zr–Al–C system.[1-3] Later, He *et al.*,[6] successfully synthesized a Hf-Al-C composite composed of $Hf_3Al_3C_5$, $Hf_2Al_4C_5$ and $Hf_3Al_4C_6$ by a hot pressing method and investigated the effect of microstructure on their mechanical and thermal properties. Therefore we aimed to fabricate single-phase ternary carbides in Hf-Al-C and Zr-Al-C systems and characterise them for their crystal structure and other properties.

Single phase $Hf_2Al_4C_5$ ceramics was fabricated from hafnium (325 mesh), aluminium (325 mesh) and graphite powders obtained from ABCR, Germany by pressure assisted sintering techniques such as hot pressing and spark plasma sintering. The processing conditions, phase analysis and microstructures were explained in previous annual report. Thin-foil specimens for TEM observations were prepared by slicing, mechanical grinding to 20 µm, dimpling down to 10 µm and ion milling at 4.0 kV. TEM observations were conducted using a 200 kV JEOL FX2100 (Tokyo, Japan) which was equipped with an EDS system, and high-angle annular dark field (HAADF) detector in scanning TEM system. Fast Fourier transformation (FFT) was carried out in the Digital Micrograph software package.

A detailed microstructural analysis was carried out by TEM. Figure 1(a) shows bright field (BF) images of $Hf_2Al_4C_5$ sintered at 1900°C by hot press. Two different types of grains are seen; large grown grains with many striations and small sub-micron size particles. Figure 1 (b) shows HAADF image of the interface between labelled regions B and D in Figure 1 (a) at a higher magnification. It can be clearly seen that large grains have very fine striations running over which represents layered structure. Figure 1 (c) is a BF image in other area. Figure 2 (a) shows again a region containing small crystals and enlarged grains. The SAED pattern (Figure 2(b)) taken on large grains along the zone axis [110] confirms it as a super layer structure. Figures 2 (c) and (d) show the interfaces of regions between B&C and C & bottom grains, respectively. Both HRTEM images show that the grain boundaries are clean and no trace of liquid is seen.

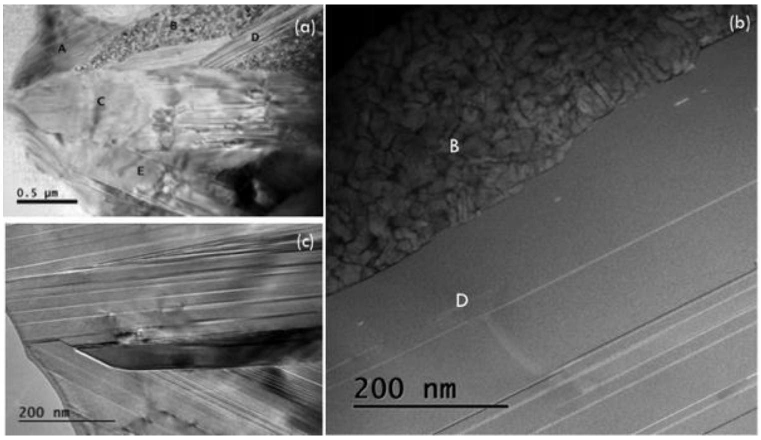


Figure 1 $Hf_2Al_4C_5$ ceramic reactive sintered at 1900°C by HP

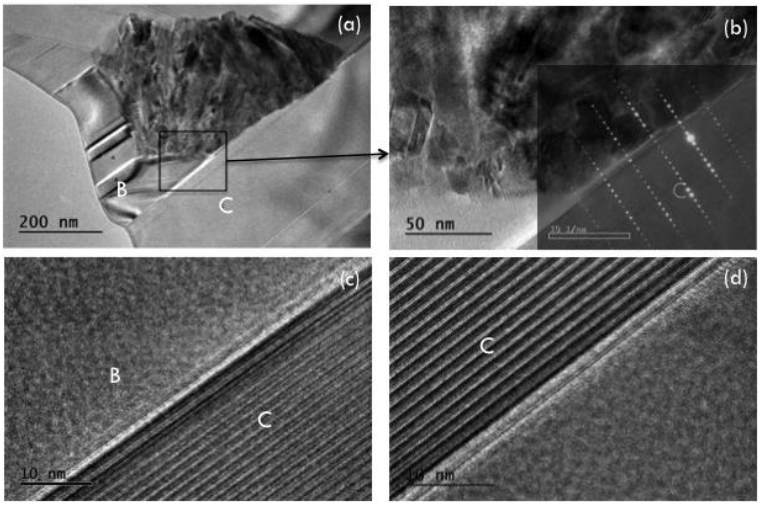


Figure 2 HRTEM image of $Hf_2Al_4C_5$ ceramic reactive sintered at 1900°C by HP

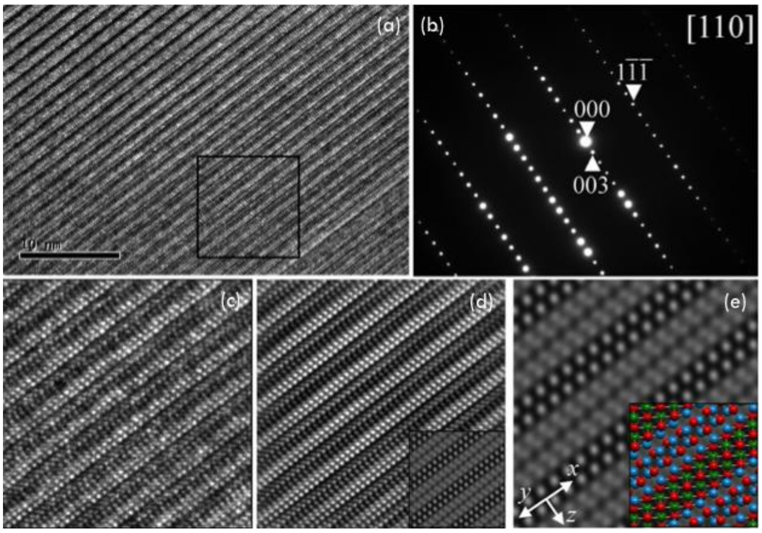


Figure 3 HRTEM, SAED, FFT and atomistic simulation of $Hf_2Al_4C_5$ –z.a.[110]

Figure 3 shows the high resolution TEM image of $Hf_2Al_4C_5$ ceramics obtained with the incident beam parallel to the [110] direction and Fig. 3(b) shows the SAED taken along the zone axis [110] and the diffraction points are indexed. Figure 3(c) shows the enlarged image of square marked in Fig.3(a). Figure 3(d) represents the corresponding FFT altered image and the inset in Fig. 3(d) shows the simulated pattern of $Hf_2Al_4C_5$ crystal using crystal maker. The simulated pattern can be nicely super imposed with FFT image. The enlarge image of FFT image can be seen in Fig.3(e). It is very clear from the FFT image and simulated image that two Hf-C slabs are separated by an Al_4C_3 layer in $Hf_2Al_4C_5$. The atomistic model shown in the inset of Fig.3(e) is created by crystal maker. The lattice parameter is calculated from the FFT image of Fig.3(d); a=b=3.082 Å, c=38.30 Å, α=β= 90°, γ=120°.

Conclusion:

TEM analysis confirms that single phase $Hf_2Al_4C_5$ was successfully synthesised by reactive sintering processing using pressure assisted sintering techniques. FFT image clearly shows that two Hf-C slabs are separated by an Al_4C_3 layer in $Hf_2Al_4C_5$.

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1. J. Wang and Y. Zhou, "Recent Progress in Theoretical Prediction, Preparation, and Characterization of Layered Ternary Transition-Metal Carbides", Annual Review of Materials Research, 39 (2009) 415-443.
2. Y.-C. Zhou, L.-F. He, Z.-J. Lin and J.-Y. Wang, "Synthesis and structure–property relationships of a new family of layered carbides in Zr-Al(Si)-C and Hf-Al(Si)-C systems", J. Euro, Ceram. Soc., 33 (2013) 2831-2865.
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6. L.F. He, Y.W. Bao, J.Y. Wang, M.S. Li and Y.C. Zhou, "Microstructure and mechanical and thermal properties of ternary carbides in Hf–Al–C system", Acta Materialia, 57 (2009) 2765-2774.
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Graphene 3D networks

Researcher : Esther Garcia-Tunon Blanca
Supervisor : Eduardo Saiz

Sponsor : EPSRC grant (EP/K01658X/1).

One of our research areas within this grant is to build graphene three-dimensional structures with practical dimensions using Additive Manufacturing (AM). AM is now considered the next industrial revolution. The goal is to produce complex 3D objects made of any possible material including nanoparticles or even 2D compounds. To address this we need to develop novel processing and post-processing approaches to broaden the AM materials palette and to optimize the structural and functional properties from the nano to the macro scale.

We have developed a new processing approach to design responsive inks for direct write assembly. We use responsive molecules[1] 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). This creates non covalent networks between the GO flakes and provides a wide range of soft-materials that can be used in several processing approaches. From molding, tape casting and emulsion templating (to create dense and porous hierarchical structures[2]), to the formulation of responsive inks with the right viscoelastic properties for filament 3D printing[3,4].

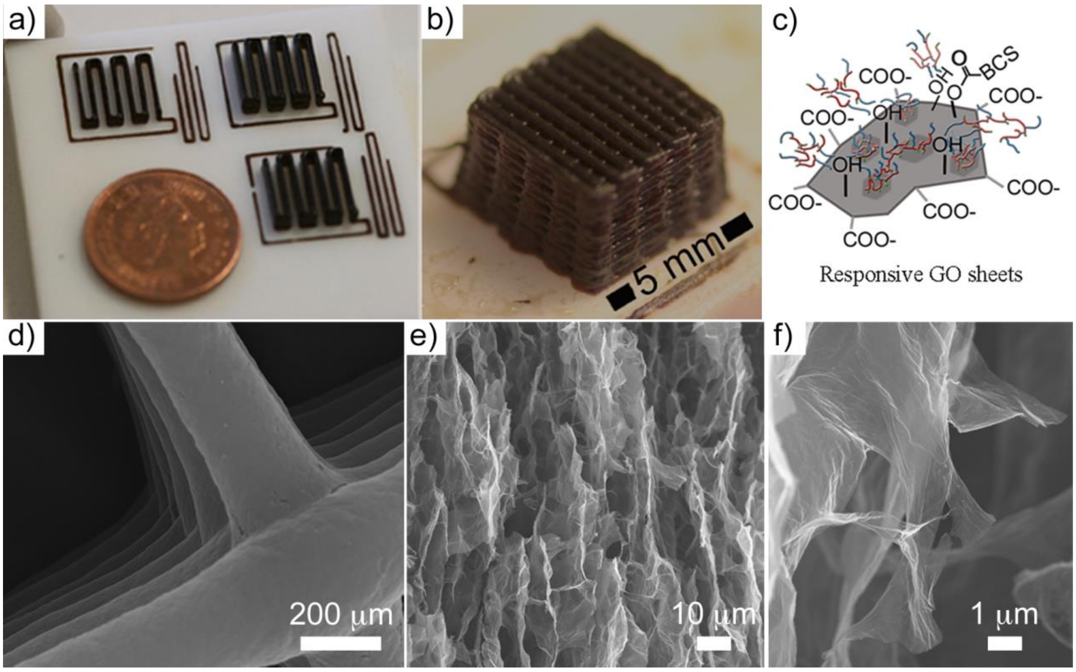

Figure 1 Using additive manufacturing to create graphene three-dimensional structures with controlled morphological features. Images a) and b) show some of the 3D printed structures in wet stage. c) Scheme of the surface functionalization of GO flakes with a responsive branched copolymer surfactant (BCS). d) Detail of a junction in a reduced graphene oxide 3D grid. e) Internal microstructure of the filaments. f) Detail of the rGO flakes.

References:

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2. García-Tuñon, D. E. *et al.* Designing Smart Particles for the Assembly of Complex Macroscopic Structures. *Angew. Chem. Int. Ed.* **52,** 7805–7808 (2013).
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4. Patent GB1410438.4: 3D printing of graphene. Filed on 11th June 2014

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.

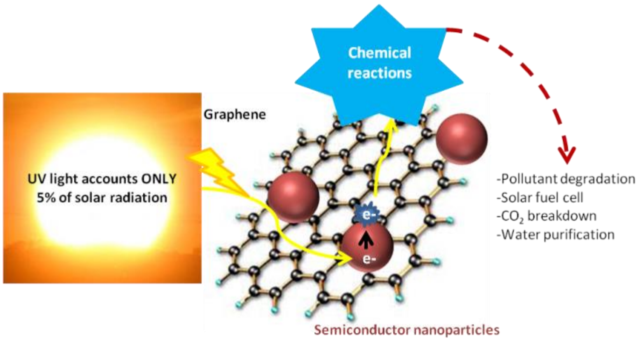


Figure 1 Schematic view of the hybrid photocatalyst working

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.

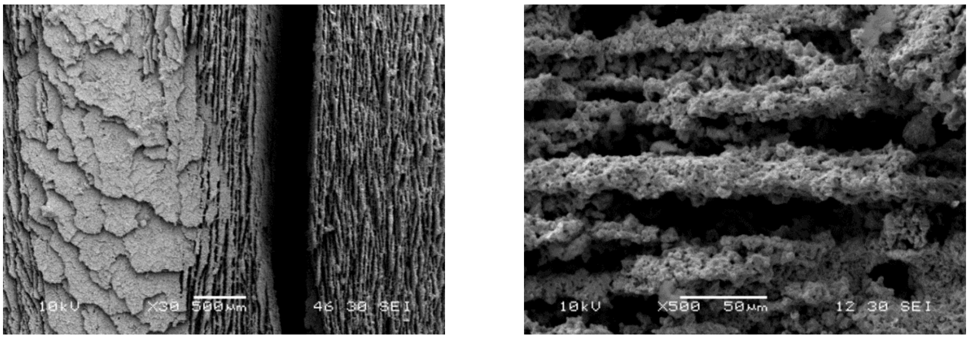


Figure 2 SEM images of freeze casted structures of $LiFePO_4$

Processing of Carbon Fibre/UHTC Composites

Researcher : Niranjan Patra
Supervisor : Doni Daniel and Bill Lee
Sponsor : Office of the Naval Research Global (ONRG), USA.

Ultra-high-temperature ceramics (UHTCs) are a family of materials with melting temperatures above 2000^{o}C which thereby have the potential to withstand extreme aerothermal heating environments. Based on the goal of developing advanced composite systems for re-entry environments, this project is working with SiC and UHTC phases such as ZrB_2, HfB_2, HfC, ZrC and TaC to create adherent, continuous coatings for enhanced oxidation protection of C–C composites. Since previous studies suggest that no single coating material would be sufficiently protective graded multilayer systems are being examined. Carbon fibre/UHTC composites are being prepared by polymer infiltration pyrolysis (PIP). Preceramic polymer solutions but not polymer-derived ceramic powder slurries are being used for infiltration.

The research is focused on UHTC infiltrated carbon fibre composites. The following objectives related to synthesis, processing, characterisation and evaluation need to be achieved:

1. Synthesis of high purity UHTC powders by wet chemical processing technique (from organo metallic precursor solution).
2. Optimization of the condition for making preceramic polymers for infiltrated pyrolysis processing (PIP).
3. Pretreatment and processing steps to create continuous and adherent high temperature ceramic coatings.
4. Developing suitable method for coating C/C composites.

Figure below shows a novel route to synthesize crystalline ZrC via polycondensation and carbothermal reduction is represented. The carbothermal reduction reaction was initiated at a lower temperature of ~1300 ^{o}C. The synthesized powders had a smaller crystallite size of ~40 nm with cubic structure.

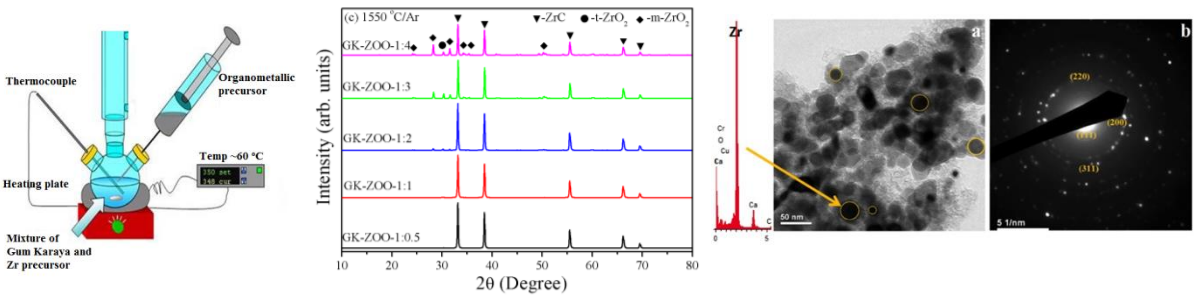


Figure 1 Schematic of the synthesis, XRD analysis of the precursor

A ZrC precursor has been developed via a low-cost process using a naturally available, biodegradable polymer source. This high carbon content polymer, Gum Karaya (GK, Sterculia urens), a tree exudate gum of natural acid polysaccharide is an excellent chelating and reducing agent which enables ZrC processing via an organometallic polymerization method at relatively low temperature (1200°C). The potential advantages of producing ZrC precursors by this route include:

- ✓ Excellent polymer infiltration and pyrolysis process for C/UHTC composites.
- ✓ Controllable viscosity by modifying the acetyl content for easy infiltration.
- ✓ Uniform and low crystallite size of the particles (~40 nm).
- ✓ High ceramic yield of more than 60 wt%.
- ✓ Low materials cost, nontoxic, less hazardous makes it an excellent route to produce ZrC.

Studying micromechanical modes of deformation in boron carbide using in-situ techniques

Researcher: Ayan Bhowmik
Supervisor: Finn Giuliani
Sponsor: EPSRC

The project broadly involves studying the modes of failure in boron carbide. Within this capacity, I am specifically interested in looking at the failure mechanism using *in-situ* microcopy, which involves both scanning and transmission electron microscopy as well as neutron and/or synchrotron diffraction techniques. Boron carbide is the preferred material for military combat armours, by the virtue of its extremely high hardness and low density. However, when subjected to high velocity impact loading, these materials fail due to the formation of localised amorphous bands. These bands create weak zones within the material that eventually lead to fragmentation. The exact mechanism of formation of these bands are hitherto disputed, so this projects will investigate the origin of the amorphous band in crystalline boron carbide subjected to loading *in-situ* electron microscopes. Addition of silicon as alloying element has been reported to eliminate the problem of amorphisation in nano-wires of boron carbides – no localised amorphisation has been observed in samples containing silicon. So the project will also investigate the possibility of fabricating Si-doped bulk boron carbide specimens and study the deformation characteristics of such materials. Currently we are trying to make diffusion bonded 'boron carbide-silicon-boron carbide' sandwich using vacuum hot pressing. Such a diffusion couple will then enable us to study the diffusion characteristics of Si in boron carbide and carry out micromechanical characterization.

Silicon doped Boron Carbide

Researcher : Yatish Patel
Supervisor : Finn Giuliani
Sponsor : Dstl

There is a need for impact resistance light-weight materials for a number of applications, for example, the aerospace industry where satellites require protection from high speed space debris or ballistic personal armour. In this case, high quality light weight armour is essential to protect soldiers and to allow them function efficiently.

In both cases high velocity impacts occur and weight is crucial. Boron carbide has the potential to be an excellent material as it is very hard and very light; however it unexpectedly fragments under shock or high pressure loading. Preliminary work has shown that by carefully adjusting the chemistry of the material with small additions of Silicon the mechanism of fragmentation under high pressure loading is suppressed, although it is unclear if this translates to improved impact performance.

My work aims to study the mechanism by which boron carbide is altered by the additions of Silicon. This will involve variations in the process methods for boron carbide and its doping with silicon. This will be followed by the resultant powder being analysed using the various material analytical techniques available such as EDS, XRD.

Development of oxidation bonded reaction sintered environmental barrier coatings on melt infiltrated SiC/SiC ceramic matrix composites

Researcher : Nasrin Al Nasiri
Supervisor : Bill Lee
Sponsor : Rolls-Royce

The need to increase the cycle efficiency and reduce noise and NO_x emissions from jet engine turbine has promoted the development of ceramic matrix composites (CMC) such as silicon carbide fibre-reinforced silicon carbide (SiC-SiC). Use of CMCs will lead to a significant improvement in fuel consumption and thrust-to-weight ratio compared to metal alloys. In addition, the low density of CMCs allows weight savings of up to 30% compared to Ni-based super alloys equating to about 1000kg/engine thus leading to vastly improved fuel consumption. However, silicon (Si)-based ceramics such as SiC-SiC have poor environmental durability in high velocity combustion environments. Si-based ceramics have excellent oxidation resistance due to formation of a protective silica layer on reacting with dry air making them stable at temperatures up to 1200˚C for long-term application. On the other hand, the same silica layer will react with water vapour to form gaseous silicon hydroxide, leading to high recession and component failure. To avoid this behaviour, a prophylactic environmental barrier coating (EBC) is required. A variety of EBCs have been developed in the past, which consists of a minimum of 4 layers requiring a costly application method such as plasma spraying. In this work, five rare earth monosilicates are being examined as potential EBCs: Y_2SiO_5, Yb_2SiO_5, Lu_2SiO_5, Gd_2SiO_5 and Er_2SiO_5. Their performance in steam environments is being studied at 1200-1350˚C for different times as a first step to determine which EBC candidate is most promising for protecting SiC-SiC CMCs in the jet engine environment. The oxidation behaviour of the SiC/SiC composites is being investigated at 900 -1300 ˚C for different times to obtain time versus temperature relationships for SiO_2 formation. Developing a low cost application method of the best EBC candidates will also be investigated. Mechanical testing will be performed at ambient conditions and at high temperatures on uncoated and coated samples as well as fatigue testing at ambient and in high-temperature steam environments.

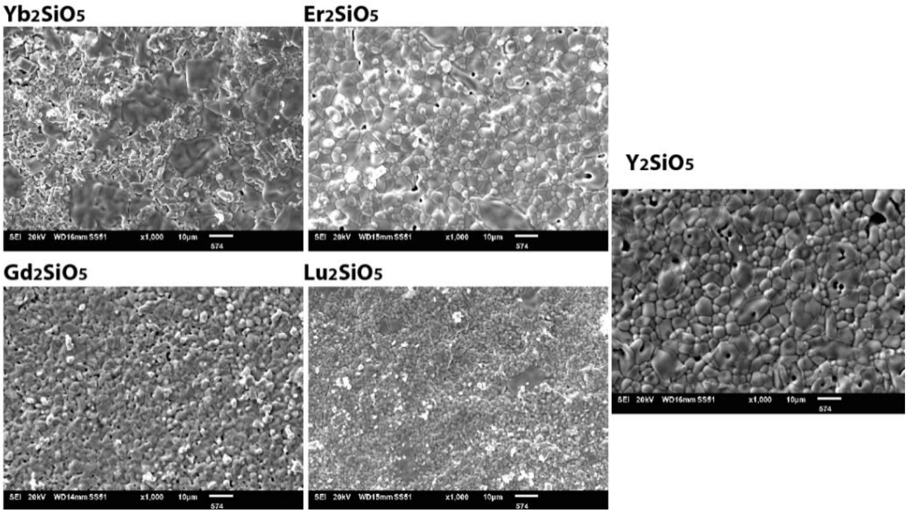


Figure 1 Scanning Electron Microscope images of the outer surface of the five rare-earth mono-silicates to be used as potential candidates for environmental barrier coating for ceramic composites. The figure shows that the five candidates have dense surfaces (no big pores are visible), which is essential to avoid oxygen diffusion through the pores.

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 thermaly reversible hydrogels that are fluid at low temperature allowing the homogeneous dispersion of ceramic particles and pseudoplastic at room temperature, allowing printing.

1. Janney, M.A., et al., *Development of Low-Toxicity Gelcasting Systems.* Journal of the American Ceramic Society, 1998. **81**(3): p. 581-91.
2. 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.

PhD Projects

Fabrication and characterisation of ZrC_xN_y ceramics as a function of stoichiometry via carbothermic reduction-nitridation

Researcher : Robert Harrison
Supervisors : Bill Lee, Robin Grimes
Sponsor : EPSRC

Currently there is a renewed interest in nuclear power as it offers an economical, low carbon solution to the planet's growing energy demand. Research interests include the processing and characterisation of non-oxides for use in Generation IV nuclear reactors with main interest focused on the gas cooled fast reactor (GFR).

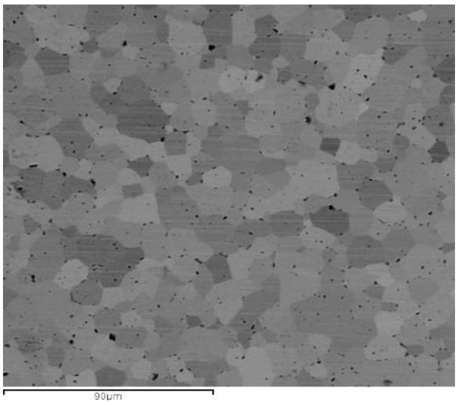


Figure 1 SEM image of $ZrC_{0.55}N_{0.4}$ ceramic

Several reference fuel concepts exist for the GFR one being Inert Matrix Fuels which consist of a dispersion of a non-oxide fissile phase (such as uranium carbide or uranium nitride) in a non-fissile material in the form of a pellet. Zirconium carbide and zirconium nitride are both promising candidates for use as the inert phase due to their high hardness, high melting point and good electrical and thermal conductivities.

The carbothermal reduction-nitridation process was used to fabricate powders of ZrC and ZrN from ZrO_2 to firstly investigate this processing route to non-oxide fuel matrices but with the additional benefit that Zr could act as a simulant for uranium or plutonium, in addition the effects that impurities, such as oxygen and carbon and vacancy defects may have on the thermophysical properties of ZrN has been examined. Combination of experimental techniques such as XRD, SEM and

TEM reveal a novel mechanism for the nitridation of ZrC. Nucleation of small ZrN containing particles occurs at the surface of the ZrC powder which grow separate to the carbide particle, resulting in a mixed phase. On annealing and densification by hot pressing the mixed phases rapidly form ZrC_xN_y solid solutions.

Room temperature thermal conductivities of the ZrC_xN_y ceramics were found to be 35 and 43 W/mK for the lowest and highest N containing ceramics produced in this work, $ZrC_{0.55}N_{0.4}$ and $ZrC_{0.12}N_{0.78}$ respectively. The thermal conductivities increased to 45 and 55 W/mK for the lowest and highest N containing ZrC_xN_y ceramics respectively at 2073 K. Room temperature electrical conductivities were in the range 250-450 × 10^4 Ω^{-1} m^{-1} for the ZrC_xN_y ceramics and again increased with increasing nitrogen content. The increase in thermal conductivity of the ZrC_xN_y ceramics with nitrogen content is attributed to the increase in electrical conductivity. The results for the highest N containing ZrC_xN_y ceramic is found to be around 4 times higher than the majority of literature data for ZrN ceramics at room temperature.

Processing and Modelling of Non-Stoichiometric Zirconium Carbide for Advanced Nuclear Fuel Applications

Researcher : Edoardo Giorgi,
Supervisors : Bill Lee, Robin Grimes
Sponsor : EPSRC

The refractory properties and high temperature properties of zirconium carbide are of great interest for nuclear fuel applications such as with the TRISO particles. As a group IV transition metal carbide ZrC exists over a wide sub-stoichiometric ratio over which its properties vary (such as conductivity). Within the life cycle of a TRISO particle the deposited sub-stoichiometric ZrC as a fission product barrier acquires carbon from the surrounding graphitic environment. It is hence important to evaluate whether this affects the effectiveness of ZrC at retaining the metallic fission products.
The research project includes a processing investigation looking into the Reactive Spark Plasma Sintering (RSPS) carbothermic route to rapidly manufacture non-stoichiometric ZrC monolithic samples. The aim of this investigation is to attempt to create a method for creating standardised samples to better aid research into diffusion. Most literature that offers data on fission product related diffusion parameters has considerable gaps and discrepancies and these can be in part attributed to the poor characterisation of ZrC samples used. Having a standardised route to produce consistent samples over a stoichiometric range is a key step to bridging the gaps in the current diffusion data.
In conjunction with the experimental study, a computational study using ab initio calculations has been undertaken to look at the nature of both intrinsic and extrinsic defects within the ZrC structure. The intrinsic defects focuses on the vacancy-vacancy interaction nature present in the sub-stiochiometric range of ZrC as ordering is known to occur within the material, however calculations have been unable to reach the size of supercells we are today capable of achieving. The extrinsic

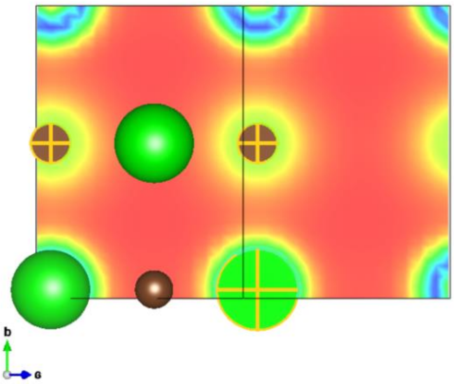


Figure 1 Charge density difference [110] plane in ZrC.

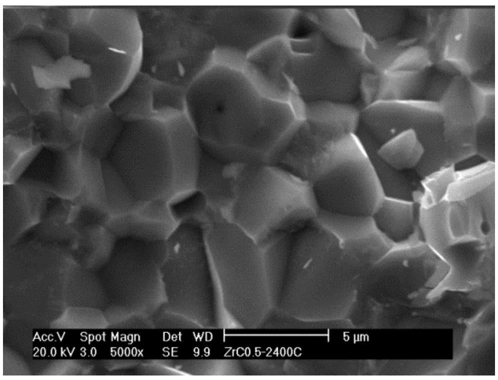


Figure 2 Reactively Spark Plasma Sintered ZrO_2+C mixture reacted into a highly dense ZrC sample.

defect side of the study focuses on the defect formation energies, defect volumes and likely diffusion paths as well as activation barrier calculations for fission products. This information will help improve our understanding of the fission product diffusion resistance of ZrC.

A Study on the Phase Evolution of Ceramic Materials Fired In Different Rapid Firing Techniques

Researcher : Wirat Lerdprom
Supervisor : Bill Lee
Sponsor : Cementhai Ceramics CO. Ltd., Thailand

It is proposed to carry out above title research project at both the Imperial Colege London and Cementhai Ceramics Company Limited. The project aims to build up an experience and knowledge in the partnership to obtain advanced and traditional microstructured of a ceramics by studying the different types of rapid firing techniques, in order to endeavor the possibility to reduce the energy consumption and develop a new of ceramic production process to produce clay based building materials.

In the last decade, tons of scientific papers and presentations have been reported and dealing with the application of the rapid sintering techniques such as Field Assisted Sintering Technology (FAST), Flash Sintering and Microwave Sintering. The rapid consolidation of powders is the most important advantage of the state-of-the-art from those firing techniques, offering significantly improved or even completed the energy consumption. The wide ranges of material types have been investigated including metals, alloys as well as Borides, Carbides, Nitrides, and Oxides, plus all imaginable composites and special materials. However, there is a few of those techniques dealing with the traditional ceramic body such Whiteware body in order to study, tailor and to obtain the final technological properties. Moreover, to study on these sintering techniques, especially in the Whiteware body, is not only to rapid consolidating the compact powder but also needed to understand how phases do evolve during the being heated. Another aspect is that most of the investigations are from scientific and academic perspectives, but are not very promising and demonstrating for a transfer to industrial production. Since, industrial production is really different from scientific point of view and requires suitable and individually tailored equipment, in order to realize a manufacturing process with optimum cost efficiency.

Since, clay based building material is composed of mainly of kaolinitic substances. The experiment is not only to densify or sinter the compact body but also, the phase evolution including metakaolin formation, mullite formation, glass phase formation and quartz dissolution have to be investigated. Thermodynamically, it could be different energy requirements to activate each reaction when a ceramic body fired in different firing techniques.

Moreover, the comparison of mathematical model and experimental testing will explain and improve the understanding of densification and phase evolution of clay-based building materials in such processes. The knowledge and result of this study will have an impact on both academic and business competitiveness. A unique formulae and production process will be providing the most efficient energy usage while promising the technological properties as similar as produced in conventional firing one.

Small Scale Fracture Testing of Polycrystalline Diamonds

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

Polycrystalline diamonds are widely employed in a number of applications primarily as tooling (e.g. for indentation, cutting, drilling, grinding, shearing and wire-drawing) typically under high stress conditions. The mechanical performance of diamond under complex stress states and in-service like conditions must be optimised to extend tool life. Currently, however, the processing parameters of such commercial composite ceramic products are largely empirically derived and therefore result in a large number of different microstructures and properties. Current methods of obtaining quantitative data for strength (i.e. toughness) and deformation mechanisms for diamond are limited as diamond is hardest and stiffest known material.

Correlating microstructures, e.g. the size and shape of diamond grains, as well as the use of binder phases in composites, with properties will enable significant improvements in tooling design and lifetime prediction, avoiding costly and problematic in-service failures and machine downtime.

This project primarily aims to develop a new fracture testing method capable of granting high spatial resolution and high control over the area to test, so to measure fracture energy of phase and interphase present in the final product.

Our approach builds upon the work of Lawn (Lawn 1993), who showed that a practical test geometry to calculate the fracture energy *G* is that of a double-cantilever beam under constant wedging displacement. We use this geometry at the small scale to directly measure fracture energy in brittle materials and small volumes.
Using Lawn's analysis, *G* is given by:

$$G = 3Eh^2d^3/4c^4$$

where *E* is the elastic modulus, *c* the crack length and *d* and *h* the half-width of the beam and the wedging displacement respectively. 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 testing approach produces data that can be analysed to directly measure the geometrical parameters required to solve the equation. Fracture behaviour investigation is then assisted by high-resolution electron backscattered diffraction (EBSD) mapping. This technique combined with an image correlation post-analysis of EBS-patterns obtained allows us to measure residual stress gradients within the grains (to a depth of ~20 nm from the surface) to better understand their influence on fracture paths. This will be the key to correlate properties to microstructure. To complete the study, information obtained will be linked to the different chemistry using TEM-EDX.

Title: Bio-Inspired Self-Healing Composites for Structural, Electrical and Sensing Applications

Researcher : Eleonora D'Elia
Supervisors : Eduardo Saiz and Theoni Georgiou
Sponsor : EPSRC

Biological tissues such as bone and nacre show remarkable properties such as high strength and toughness and the ability of self-repair. To a large degree the unique performance of these natural materials is due to the presence of thin, interfacial organic layers separating stiff bricks. 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 use Double Cantilever Beam (DCB) tests of 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.

Furthermore, we use the same self-healing organic phase to develop an innovative method for the production of skin-like composite films, able to flex and completely and autonomously repair via hydrogen and dative bonds. These materials, obtained by the careful infiltration of graphene ultra-light cellular networks with the self-healing polymer, show repair times of the order of a few minutes, where the "skin" recovers its flexibility just by putting the two fractured surfaces back into contact. The material is able to repair scratches and superficial damage as well as deep tissue tear. Moreover, it presents the properties of an electrical sensor by varying its conductivity under applied pressure or flexion. The chemistry of the graphene aerogels has been tailored to achieve excellent adhesion with the organic phase. The continuous graphene network provides structural integrity, strength and electrical conductivity while keeping the graphene contents below 0.5 wt.%. We have characterized the mechanical strength, viscoelastic response and electrical properties of the material. Tensile strengths of the order of 0.2 MPa are combined with conductivities that can be as high as hundreds of S/m. Both can be recovered after damage in a few minutes. We therefore show here how it is possible to mimic key properties of skin via a synthetic route to create a strong and flexible electronic sensor able to autonomously self-heal without the use of pressure or heat.

Development of a Novel Wound Management Dressing

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

Bio-inspired ceramic-based composites

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

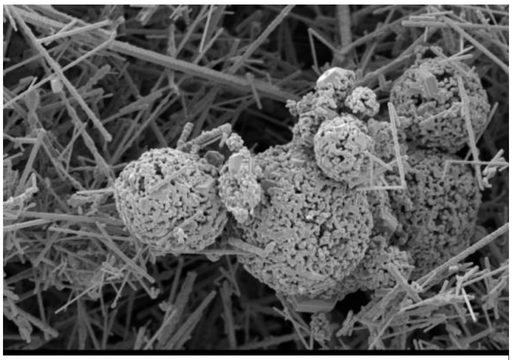


Figure 1 CVD deposition of SiC spheres during the sintering at high temperatures of SiC scaffolds

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 $MPa\cdot m^{1/2}$) 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.

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

Calcium phosphate ceramics are widely considered appealing materials for the repair of bone defects, because of their similar composition to the inorganic phase of natural bone. Two of the most commonly investigated calcium phosphates are Hydroxyapatite (HA) and β-Tricalcium phosphate (β-TCP), which are generally designated as Biphasic Calcium Phosphates (BCPs) when used in combination. However, despite the development of many different processing routes, there is still a clear need of adequate techniques to create calcium phosphate ceramics with controlled chemistry and structural features.

Evidence shows that surface topography in the micrometre range plays a crucial role in the modulation of cellular response to biomaterials. Our main objective is to develop a framework to understand the interplay between chemistry and surface topography that will lead to the design of optimised calcium phosphate scaffolds with complex architectures. To achieve this goal we need to develop methods for the reproducible and practical preparation of large 3D scaffolds with controlled structural features at different length scales for the treatment of bone defects, through various shaping techniques – including 3D-printing.

A novel manufacturing process to fabricate calcium phosphate structures and surfaces with controlled architectures based on the design of responsive particles was developed. The method uses a pH-responsive branched copolymer surfactant (BCS) to functionalize surfaces in situ and to create responsive particles that can assemble between themselves or with soft templates (i.e. in emulsified suspensions) under the action of an external trigger (pH). The system enables a tight control of the viscoelastic properties of the ceramic slurry, and can be used in additive manufacturing technologies to obtain materials with complex shapes and tailored microstructure at different length scales. The structures can mimic to some extent the hierarchical architecture of natural materials such as trabecular bone, opening new paths for the optimization of their mechanical and biological performance.

Using this technique, we have been able to fabricate 2-dimensional substrates with controlled surface topography and chemical composition and are on the way to completing their biological characterization using Human Bone Marrow-derived Mesenchymal Stem Cells. This work was performed at the AO Research Institute in Davos, Switzerland as part of the BioBone-ITN protocol.
The work so far has led to some very promising results. Figure 1 shows how the microstructure obtained from the soft-templating with the particle-stabilized emulsions is kept when used in 3D-printing of complex shapes.

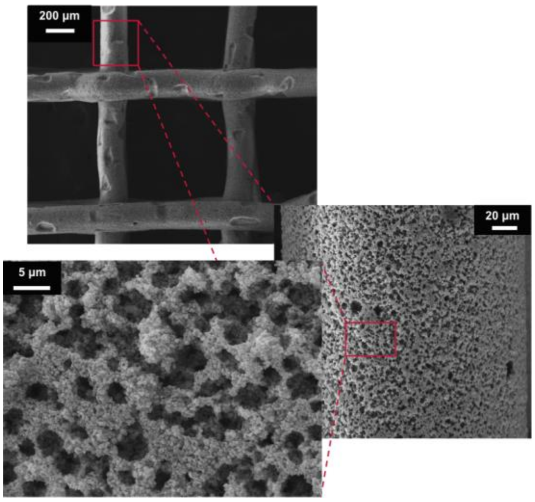


Figure 1 SEM micrographs of 3D printed HA (70 wt%) functionalized with BCS

In Figure 2, one can see the morphology of a hBMSC on a HA rough substrate after 48h of incubation. It is clear that it adheres to the material, interacting with it through many cellular extensions (pseudopodia).

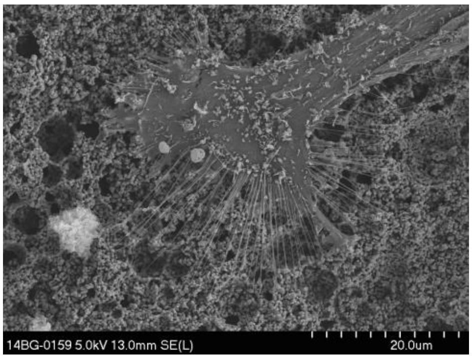


Figure 2 SEM micrographs of 3D printed HA (70 wt%) functionalized with BCS emulsions

Robocasting of Functionally Graded Ceramic Materials

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

The additive manufacturing technique “Direct Ink Writing” or “Robocasting” has shown great promise over the last two decades for the production of geometrically complex 3D scaffolds using a variety of materials including ceramic, metallic, glass and polymeric powders. A number of different “inks” have been designed and developed to facilitate deposition of these materials, many of which are capable of carrying virtually any powder. The behaviour of the ink is crucial to the quality of the fabricated parts, influencing structure from the micron to the meter scale.

There is a great need to establish this technique’s ability to produce ceramic bodies with varying shapes and densities with the inks currently available. In particular, it is not known whether it is possible to produce parts with mechanical properties comparable to those fabricated by traditional methods.

Thus far, tool path, nozzle speed and size, ink rheology, printing environment and sintering parameters have been optimised with SiC based and Alumina based inks to produce simple dense solid parts (densities >95th%) as well as designs from arbitrary CAD files. A number of different ink formulations have also been produced and their rheology compared.

Robocasting has the potential to form intricate functional grading within parts with resolutions <100 µm. The orientation of anisotropic additives such as short fibres can be manipulated during printing and porosity and composition can be varied by co-depositing multiple inks. This will be the primary area of future research. A number of different ink formulations will be tried but particular attention will be paid to hydrogel-based inks due to their scaling-up potential and industrial applicability.

Lastly, after acceptable dense, porous and functionally graded bodies have been shown to be achievable with silicon carbide, attention can shift onto printing geometries which could exploit these features for a number of applications. One application of interest is leading edge geometries for atmospheric re-entry vehicles. Mesoscale structures such as cooling channels and heat pipe geometries could be printed. Porosity could be controlled on multiple scales to optimise heat pipe performance, and fibres could be printed in tailored structures to maximise thermal shock resistance. The final stage of the project will be the testing of these prototype infiltrated heat pipe parts at room and high temperatures and exposure to re-entry like conditions.

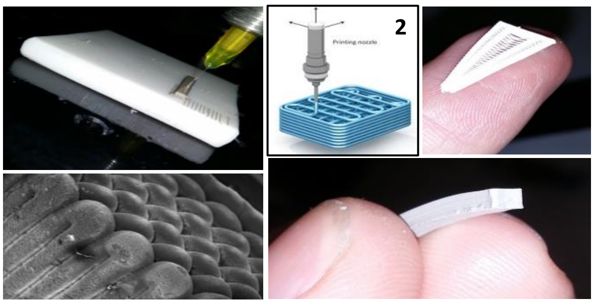


Figure 1 A- Printing with a silicon carbide pluronic ink through a 100um nozzle. B- Schematic of the Robocasting process. C- Unsupported alumina test structure. D- Micrograph showing a staggered raster fill pattern of a sintered bar. E- Green silicon carbide bar with <5% residual porosity after sintering.

Durability of Synroc-type Nuclear Waste Materials

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

Synthetic Rock (Synroc) was developed in Australia in the 1970’s as a waste form capable of hosting wide range of radioactive waste. The Australian Nuclear Science and Technology Organisation (ANSTO) has been working with the National Nuclear Laboratory and Sellafield Ltd. in the UK to develop Synroc compositions for some of the UK's plutonium contaminated waste. Synroc is possible to tailor the matrices for radioactive wastes and also durable to stand in geological environments. It is one of the suitable waste forms to immobilize High Level Waste (HLW) from advanced spent fuel reprocessing processes.

My research will characterize simulant and actual Synroc waste forms and examine their durability in a range of likely repository groundwater types using both empirical and modelling approaches.

Characterisation techniques including XRD, SEM, FIB, TEM and SIMS will be used to examine the as-processed microstructures which will also be characterized after corrosion testing in aqueous environments.

Resource efficient reuse applications for recycled glass and Wastepaper Sludge Ash (WPSA)

Researcher : Charikleia Spathi
Supervisors :Luc Vandeperre, Chris Cheeseman
Sponsor : EPSRC Industrial Case Award, Smithers PIRA

After graduating from the National Technical University of Athens in Greece with a MEng in Chemical Engineering, I completed a Master's in Environmental Engineering and Business Management at Imperial College London (ICL). I am currently in the fourth year of my PhD, which is funded by EPSRC and Smithers PIRA, under the supervision of Professor Chris Cheeseman and Dr Luc Vanderperre at ICL. The key industrial collaborators are Aylesford Newsprint Ltd. and UPM-Shotton.

The object of my research is to develop novel, higher-value reuse applications for recycled glass and wastepaper sludge ash (WPSA), with the latter one being the residue from the combustion of wastepaper sludge. My research activities focus on the production of artificial lightweight filler (LWF) materials with enhanced physical, mechanical and thermal insulating properties. Key findings of ongoing research indicate that a glass-WPSA system can form foamed materials using simple processing technology involving wet milling, pelletising and low temperature sintering. Therefore, there is significant potential for the artificial glass-WPSA LWFs to substitute depleting naturally sourced fillers used in construction materials

Beneficial reuse of the problematic fraction of incinerator bottom ash

Researcher: Athanasios Bourtsalas
Supervisors: Chris Cheeseman, Sue Grimes, Luc Vandeperre
Funded by: Martin Gmbh, Wheelabrator Technologies Inc., Global WTERT Council

My research focuses on the processing necessary to transform the problematic fine fraction of incinerator bottom ash (IBA) into a raw material for manufacturing ceramic tiles. IBA is produced by energy from waste (EfW) facilities processing residual municipal solid waste (MSW) and is extensively processed to extract ferrous and non-ferrous metals and produce high quality secondary aggregate for civil applications. However this processing also produces significant volumes of fine IBA with particle sizes typically less than 4 mm which is problematic because reuse options for this fraction are limited. This work reports on the manufacture of ceramic powders from this problematic fine fraction of IBA that are suitable for the production of sintered ceramics tiles via conventional uniaxial pressing and sintering. Processing of the problematic IBA fraction involved the addition of waste glass, milling to reduce particle size and increase reactivity and drying. The powder formed is then calcined to reduce shrinkage during sintering and this transforms the original crystalline phases present into a processed powder containing diopside ($CaMgSi_2O_6$), clinoestatite ($MgSiO_3$) and andradite ($Ca_3Fe_2Si_3O_{12}$). The optimised powder exhibited low shrinkage (< 5%) during sintering of pressed tiles and produced a high quality dense (2.78 g/cm^3) hard ceramic that exhibit negligible water absorption. The research demonstrates the potential for transforming the problematic fraction of IBA into a raw material suitable for the manufacture of ceramic tiles for urban applications.

Silicon doped boron carbide as a lightweight impact resistant material

Researcher : Cyril Besnard
Supervisors : Luc Vandeperre and Finn Giuliani
Sponsor : Defence Science and Technology Laboratory

The project is done in collaboration with the Defence Science and Technology Laboratory of the UK. The aim is to develop ceramics for use in armour. Lightweight ceramics are attractive for a number of applications. For the personal armour, the ceramic has to deflect the projectile and absorb the energy; be superior to the threat. Low density and high hardness are crucial for this application. B_4C is an ideal candidate and has already been used since many decades. However the crystalline structure collapses at high velocity. Research done to improve B_4C such as doping with silicon could be a way to improve the phase stability of the ceramic. Hence, this is being investigated in this project.

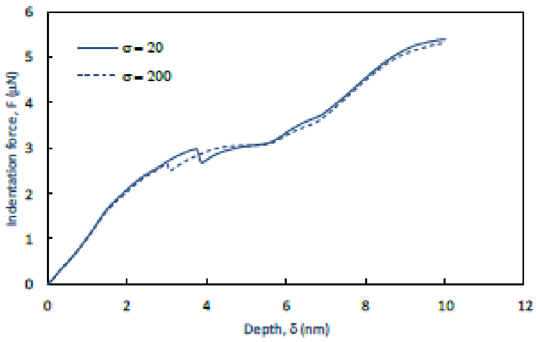

Roland

Capabilities and facilities

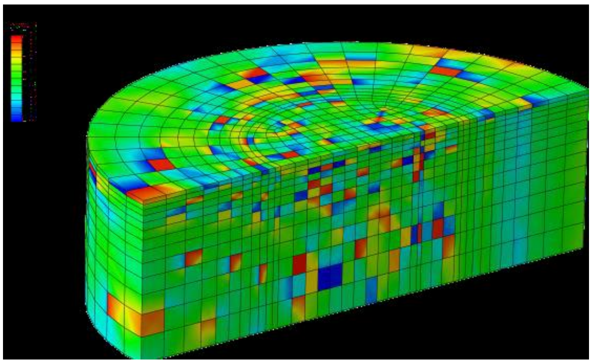
The purchasing and installation of large items of equipment by the Centre from the original CASC project, to improve UK capability in fabrication and modelling of structural ceramics, is now complete but we continue to improve our experimental capability in this area using funds from other sources. All 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).



Server



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.

Induction-heated thermo-mechanical test rig

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)

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

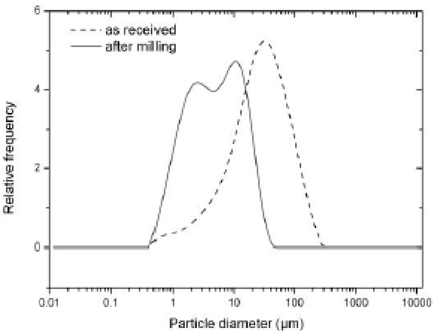
The vacuum furnace from Thermal Technology is now fully operational. The furnace will heat a volume 5 cm diameter and 15 cm tall to temperatures up to 2500 °C under vacuum or a mixture of gasses. Opposed viewing ports will allow observation of the sample during heating, and a sample elevator and cooling 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

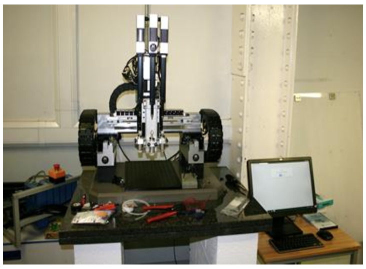
High Temperature elastic properties by impulse excitation

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

Other equipment

Other equipment like a new polishing machine and glove box were installed in CASC in 2014.

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

An Oxygen-nitrogen-hydrogen Elemental Mass Gas Analyser (EMGA series) has been placed in order and will be 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 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.

CASC Research Portfolio

Funded proposals (Jan – Dec: 2014)

- Bill Lee, Nuclear Energy Centre for Doctoral Training, 2014-19, with Cambridge and The Open University, £4M (£3M to Imperial)
- Bill Lee and Doni Daniel, Improving Oxidation Resistance of SiC/SiC, Rolls-Royce, £265 K
- Bill Lee, Synroc Durability PhD, University of New South Wales, Australia, 2014-18, £180k
- Bill Lee, EPSRC Nuclear Decommissioning Authority Industrial CASE, 2014-18, £150 K
- Bill Lee, Lloyds Register, Support for Chair in Nuclear Regulation, 2014-18, £150 K
- Eduardo Saiz, Novel Wound Management Dressing, Welland Medical, £50K.
- Eduardo Saiz, Graphene Composites for Pipelines, Petronas, 1/8/ 2014-30/7/ 2017, £400K
- Finn Giuliani and Ben Britton, TEM of Injector deposits, Shell, £98K
- Finn Giuliani, Mary Ryan, Alexander Porter, Ben Britton and Payne, Shell £1.22 M
- Luc Vandeperre, Exchange of radions on cement components, AMEC, £80k
- Luc Vandeperre, Effluent treatment with functionalised nanoparticles, EPSRC/NNL/NDA/Sellafield, £80k

Publications (Jan – Dec: 2014)

Books and Book Chapters

- Lee WE, Harrison R, Giorgi E, Maitre A and Rapaud O, Fahrenholtz WG, Wuchina EJ, Lee WE and Zhou Y N(Eds.) Nuclear Applications for Ultra-High Temperature Ceramics and MAX Phases, in Ultra-High Temperature Ceramics: Materials for Extreme Environment Application, (Wiley 2014).
- William G. Fahrenholtz, Eric J. Wuchina, William E. Lee and Yanchun Zhou (Eds.) UHTC composites for hypersonic applications, A Paul, J Binner and B Vaidhyanathan, Chapter 7, in Ultra-High Temperature Ceramics: Materials for Extreme Environment Applications, First Edition. The American Ceramic Society. Published by John Wiley & Sons, Inc, 144-166, (2014).
- J Wang, L.J. Vandeperre, Deformation and Hardness of UHTCs as a Function of Temperature, in Ultra-high temperature ceramics : Materials for extreme environment applications, eds. Fahrenholtz, W.G., Wuchina, E.J., Lee, W.E., Zhou, Y., John Wiley & Sons, Hoboken, (2014), 236-266.
- MI Ojovan and WE Lee, An Introduction to Nuclear Waste Immobilisation, (2^{nd} Edition, Elsevier 2014) 362.

Journal Papers (Jan – Dec: 2014)

- Flash Spark Plasma Sintering (FSPS) of Pure ZrB2 Powder, Grasso S, Saunders T, Porwal H, Cedillos-Barraza O, Jayaseelan DD, Lee WE and Reece M, J. Am. Ceram. Soc. 97 [8] 2405-2408 (2014).
- Effect of La_2O_3 Addition on Long-term Oxidation Kinetics of ZrB2-SiC and HfB2-SiC Ultra-high Temperature Ceramics, Zapata-Solvas E, Jayaseelan DD, Brown PM and Lee WE, J. Euro. Ceram. Soc. 34 [5] 3535-3548 (2014).
- Thermophysical characterisation of ZrCxNy ceramics fabricated via carbothermal reduction-nitridation, R Harrison, O Ridd, DD Jayaseelan and WE Lee, J. Nuclear Mats. 454 (1-3) 46-53 (2014).
- Effect of Oxidation on Mechanical Properties of Ultra-high Temperature Ceramics, (accepted), Zapata-Solvas E, Jayaseelan DD, Brown PM and Lee WE, J. Euro. Ceram. Soc. (2014).
- Synthesis of biopolymer-derived zirconium carbide powder by facile one-pot reaction, N. Patra, D. D. Jayaseelan, and W. E. Lee, J. Am. Ceram. Soc., 1–7 (2014), DOI: 10.1111/jace.13321.
- Fabrication of ZrCxNy from $_{ZrO2}$ via two step carbothermic reduction-nitridation, R. Harrison, O. Rapaud, N. Pradeilles, A. Maitre, W.E. Lee, Journal of the European Ceramic Society, In Press (2014).

- Microstructural evolution of HfB_2 - based ceramics during oxidation at 1600 - 2000°C, D.D. Jayaseelan, E. Zapaa-Solvas, C. Carney, A. Katz, P. Brown and W.E. Lee, Advances in Applied Ceramics (accepted).
- Development of multi-layered thermal protection system (TPS) for aerospace applications, D.D. Jayaseelan, X. Yanda, L. Vandeperre, P. Brown and W.E. Lee, Composite: Part B (accepted).
- Flash spark plasma sintering (FSPS) of pure $ZrB_{2,}$ S. Grasso, T. Saunders, H. Porwal, O. Cedillos-Barraza, D.D. Jayaseelan, W.E. Lee and M.J. Reece, J. Am. Ceram. Soc., 97(8), 2405-2408 (2014).
- Thermal properties of La_2O_3-doped ZrB_2 and HfB_2-based ultra-high temperature ceramics, E. Zapaa-Solvas, D.D. Jayaseelan, P. Brown and W.E. Lee, J. Eur. Ceram. Soc., 33, 3467-3472 (2013).
- Mechanical properties of ZrB_2 and HfB_2 based ultra-high temperature ceramics fabricated by spark plasma sintering, E. Zapaa-Solvas, D.D. Jayaseelan, P. Brown and W.E. Lee, J. Eur. Ceram. Soc., 33, 1373-1386 (2013).
- Enhanced Oxidation resistance of ZrB_2/SiC Composites through In-situ Reaction of Gadolinium Oxide in Patterned Surface Cavities, (submitted), J Gonzalez-Julian, O Cedillos, S Doring, S Nolte, O Guillon and WE Lee, J. Euro. Ceram. Soc.
- Encapsulation of aluminium in geopolymers produced from metakaolin, Kuenzel, C., Neville, T.P., Omakowski, T, Vandeperre, L.J., Boccaccini, A.R., Bensted, J. Simons, S.J.R., Cheeseman, C.R., J. Nucl. Mat. 447(1-3), (2014), 208-214.
- Formation of magnesium silicate hydrate (MSH) cement pastes using sodium hexametaphosphate Zhang, T., Vandeperre, L.J., Cheeseman, C.R., Cem. Concrete Res. 65, (2014), 8-14
- Recycling disposable cups into paper plastic composites, Mitchell, J., Vandeperre L.J., Dvorak, R., Kosior, E., Tarverdi, K., Cheeseman, C.R., Waste Management 34(11), (2014), 2113-2119
- Influence of sand on the mechanical properties of metakaolin geopolymers, Kuenzel, C., Li, L., Vandeperre, L.J., Boccaccini, A.R., Cheeseman, C.R., Constr. Build. Mat. 66, (2014), 442-446.
- High Temperature Characteristics of Refractory Zirconia Crucibles Used for Vacuum Induction Melting, A Quadling, L. Vandeperre, P. Myers and W.E. Lee, pp 102-107 in Proc. Unified International Technical Conference on Refractories, UNITECR '13, Victoria, British Columbia, Canada, 2014.
- Microstructure Evolution in Calcium Aluminate cement Bonded Castables at High Temperatures, J Alex, L Vandeperre, B Touzo, C Parr and WE Lee, Effect of Sodium Impurities on Phase and, pp. 832-837 in Proc. Unified International Technical Conference on Refractories, UNITECR '13, Victoria, British Columbia, Canada, 2014
- Mesoscale assembly of chemically modified graphene into complex cellular networks, Barg, S., F.M. Perez, N. Ni, P.D.V. Pereira, R.C. Maher, E. Garcia-Tunon, S. Eslava, S. Agnoli, C. Mattevi, and E. Saiz,. Nature Communications, 5 2014.
- Interfacial energies and mass transport in the Ni(Al)-Al_2O_3 system: The implication of very low oxygen activities, Ni, N., Y. Kaufmann, W.D. Kaplan, and E. Saiz,. Acta Materialia, 2014. 64: 282-296.
- Macro porous polymer nano composites synthesised from high internal phase emulsion templates stabilised by reduced graphene oxide, Wong, L.L.C., S. Barg, A. Menner, P.D. Pereira, G. Eda, M. Chowalla, E. Saiz, and A. Bismarck,. Polymer, 55(1): p. 395-4022014.
- Bioinspired structural materials, Wegst, U.G.K., H. Bai, E. Saiz, A.P. Tomsia, and R.O. Ritchie,. Nature Materials, 2014. doi:10.1038/nmat4089.
- An investigation of the mineral in ductile and brittle cortical mouse bone. Journal of Bone and Mineral Research, Rodriguez-Florez, N., E. Garcia-Tunon, Q. Mukadam, E. Saiz, K.J. Oldknow, C. Farquharson, J.L. Millan, A. Boyde, and S.J. Shefelbine, 2014. DOI: 10.1002/jbmr.2414.
- Printing in three dimensions with grapheme, Garcia-Tunon, E., S. Barg, J. Franco, R. Bell, S. Eslava, E. D'Elia, R.C. Maher, F. Guitian, and E. Saiz,. Advanced Materials, 2014 accepted.

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. A newsletter was circulated in September 2013, covering additional CASC research and equipment, visitors to the Centre and the second summer school on ceramics.
(https://workspace.imperial.ac.uk/structuralceramics/Public/Newsletter%205%20for%20CASC%202013.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.

Imperial Festival 2014

Record numbers of visitors joined staff and students for the annual celebration of the best science and arts on offer from Imperial College London. May 8-10, more than 12,000 public and alumni visitors descended on Imperial's South Kensington campus to enjoy the interactive, workshops, tours, talks and performances on offer at the 2014 Imperial Festival.

The CASC research activities were showcased in the festival. A flyer explaining the scope and aim of the CASC industrial consortium was published for public awareness.

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 Ceramics. 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 2013 prize was won by Dr Huixing Zhang. Huixing's research on *Microstructural Evolution and Oxidation Behaviour of Spark Plasma Sintered MAX Ceramics* was supervised by Professor Ping Xiao at University of Manchester. The award in general covers a certificate, plaque and £1000 cheque.

CASC Industry Day

Fourth CASC Industry Day has been organised for 17 January 2014 at Imperial College with attendants from industry (Morgan Advanced Materials, Rolls-Royce, Ceram Tec, Dynamic Ceramic, Advanced Defence Materials Ltd., Office of Naval Research Global, UK, Atomic Weapons Establishment) and university (Imperial, Queen Mary, Missouri Rolla University of Science and Technology). There were presentations and discussions between industrial and academic members as well as research posters. The 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 5th edition of CASC's Ceramic Sumer School took place from 17 to 19 September 2014. The school consisted of (i) introductory lectures on forming, sintering and mechanical properties, (ii) laboratory experiments on powder characterization, colloidal suspensions and mechanical properties, iii) master classes by experts: Dr Esther Garcia-Tuñon Blanca from CASC on additive manufacturing, Dr David Pearmain and Dr Aminat Bolarinwa from Lucideon on scaling up field enhanced sintering for industrial application, Dr Annelies Malfliet (KU Leuven) on refractories for the copper industry and Prof John Fernie (AWE) on joining of ceramics. The attendees came mostly from from industry and/or government labs (89%) and 94% would recommend the school to a colleague, especially if in need of a refresher or relatively new to the field. The 6th edition will take place from 16 to 18 September 2015.

Friday's 'al-fresco' lunch

Lab session on colloids

CASC seminars

The following seminars were arranged in CASC during 2014

20 Feb. 2014	Damage and fracture evolution in ceramic coatings under thermal shock Prof Vadim V. Silberschmidt Head, Mechanics of Advanced Materials Research Group Loughborough University, Loughborough
13 Mar. 2014	Dental Enamel: Lessons from a Hierarchical Composite Professor Van P Thompson Biomaterials,Biomimetics and Biophotonics, King's College London Dental Institute, UK
10 April 2014	Probing the local chemistry of 2D materials and oxides Rebecca J Nicholls Department of Materials, University of Oxford,
10 July 2014	Molecularly-tailored nanomaterials and interfaces: untangling and enhancing unfavorably coupled properties Prof Ganpati Ramanath John Tod Horton Professor of Materials Science and Engineering Rensselaer Polytechnic Institute, Troy, NY 12180, USA