

# **Centre for Advanced Structural Ceramics 2018 Annual Report**

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Cover image by Dr Ezra Feilden Irving.

# INDEX

Introduction.....	p: 1
Management.....	p: 2-4
Local Management Team	
Industry Consortium Group	
People .....	p: 5-9
Staff	
Researchers	
PhD Students	
Academic Visitors	
CASC Alumni	
Research.....	p: 10-29
PDRA Projects	
PhD Projects	
Capabilities and Facilities.....	p: 30-38
CASC Research Portfolio.....	p: 39-43
Funded Proposals	
Publications	
Outreach.....	p: 44-46
Newsletter	
Website	
Sir Richard Brook Prize	
CASC Industry Day	
CASC Summer School	
Conferences	

## Introduction

This has been a very special year for CASC. We just had our 10-year anniversary as CASC started running in July 2008 with EPSRC funding (£5.5M).

The original idea was to be a five-year programme, but we have been running an extra 5 years, as the EPSRC funding came to an end in June 2013. The secret for this has been to establish new industrial collaborations from abroad and in the UK, together with different research projects.

We have another motive for our happiness, CASC is growing! We now have two new lecturers that joined us in October 2018 and Dr Vandeperre is now Professor Vandeperre.

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

Our main goal is to continue these relationships and grow as a ceramics centre.



**Professor Eduardo Saiz**

**CASC Director**

## **MANAGEMENT**

CASC was initially set up by Professor Bill Lee in 2008. In 2012, he was succeeded as Director by Professor Eduardo Saiz.

### **Local Management Team (LMT)**

The LMT is responsible for managing the centre's operations and meets the second Thursday of every month to oversee the pressing day-to-day issues of running the Centre. These issues include staff appointments, equipment purchase, finances and building refurbishment, but are increasingly focussed on developing the Centre national and international profile, forging industrial links and achieving financial sustainability. This meeting also gives the chance to PhD students and Postdoctoral researchers to discuss important matters for them and for the people in the office,

The LMT is chaired by Eduardo Saiz and other members are Finn Giuliani, Luc Vandeperre, Florian Bouville, Katharina Marquardt Ainara Aguadero, Stephen Skinner, and Garry Stakalls.

The meetings are also attended by representatives of the Postdoctoral Researchers (Dr Iuliia Elizarova) and PhD students (Ms Jia Hui Teo) 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.

After the end of the EPSRC funding in 2013, an industrial consortium scheme was set up to build on CASC's early success, to enable its sustainability and to support the long-term and fruitful relationships created between CASC-associated academics and UK's industry. This was planned and presented in our first Industry Day meeting the 17<sup>th</sup> of May of 2011, where it was well received by the industry representatives and was developed by our Steering Group on July 4<sup>th</sup> 2011.

The Industrial Consortium started functioning in 2014 after the Steering Group meeting, held the 17<sup>th</sup> of January 2014.

The ICG develops the CASC Business Plan which contains the Centre's vision, objectives and an action plan to deliver such vision. It also acts as an advisory role to the Director and the Local Management Team, 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 (table in page 4).

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. On the other hand, Sapphire and Ruby memberships are aimed at companies who want to collaborate with CASC on research and training.

All three levels of membership provide:

- Access to CASC equipment, at preferential rates, (including hot press, vacuum furnace, Nano-indenter...) with operator and interpretations.  
The degree of access will depend on the level of membership as seen in the table below.
- Access to CASC and CASC associated academics.
- A number of free positions at CASC 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 and descriptions of such are needed by Easter previous year.
- Opportunity to propose research projects for students on Master 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 subject for a PhD funded by the consortium.
- To date we have 1 member signed up at Sapphire level (Morgan Advanced Materials) and 4 members at Ruby level (Asahi Glass, SAFRAN, Reaction Engines and John Crane) and we 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](mailto:e.saiz@imperial.ac.uk) – 020 7594 6779> or Alba Matas Adams < [a.matas-adams@imperial.ac.uk](mailto:a.matas-adams@imperial.ac.uk) – 020 7594 2053>

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

## **PEOPLE**

### **Staff**

#### **Professor Eduardo Saiz**



Eduardo has been CASC's Director since August 2012. He previously was a Staff Scientist at the Materials Sciences Division of Lawrence Berkeley National Laboratory (LBNL) and joined CASC in October 2009. Eduardo took over the role of Deputy CASC Director in July 2010.

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 and 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 his 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 was the principal investigator of the EPSRC award. Bill is a Professor of Ceramic Engineering and Co-Director of the Institute for Security Science and Technology at Imperial College. His research covers processing-property-microstructure relations in refractories, whitewares, nuclear and ultra-high temperature ceramics. Bill was made a Fellow of the Royal Academy of Engineering in 2012, was President of the American Ceramic Society from Oct 2016 to Oct 2017 and became a Foreign Fellow of the Indian National Academy of Engineering in 2017.

#### **Dr Finn Giuliani**



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

Finn has a PhD from the University of Cambridge where he examined small scale plasticity in multi-layered 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  $\text{Ti}_3\text{SiC}_2$ , 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.

The focus of the majority of his research at this time is small scale mechanics particularly stable small scale fracture experiments. These allow the properties of interfaces and grain boundaries to be measured directly.

### **Dr Luc Vandeperre**



Dr Luc Vandeperre, currently a reader in the Department of Materials, joined the CASC academic staff in July 2010. He is currently the Deputy Director of CASC and ICO-CDT Director of the Centre for Doctoral Training in Nuclear Energy.

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 thermo-mechanical 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 Florian Bouville**



Dr Florian Bouville joined the Department of Materials and the Centre for Advanced Structural Ceramics as Lecturer in October 2018.

Before that, he obtained his Master's degree in Material Sciences at the Institut National des Sciences Appliquées de Lyon (INSA de Lyon, France) in 2010. He then moved to the South of France for his PhD between three partners: the company Saint-Gobain, the Laboratory of Synthesis and Functionalization of Ceramics and the MATEIS laboratory (INSA de Lyon). His research was based on the freezing of colloidal suspensions and

self-assembly to process bio-inspired materials. From 2014 to 2018, he was a postdoctoral researcher and then scientist in the Complex Materials group of Prof. André R. Studart at the

Department of Materials at the ETH Zürich. His research field is mainly on new additive manufacturing processes for inorganic materials, with an emphasis on toughening mechanisms and functional properties of architected ceramics.

### Dr Katharina Marquardt



Katharina joined the Department of Materials in October 2018 as a Lecturer in Ceramics. Prior to moving to Imperial College, she worked at the University of Bayreuth at the Bayerisches Geoinstitut. She received a doctorate from the Technical University Berlin for a collaborative effort with the GeoForschungsZentrum Potsdam. As visiting researcher, she spent time at the National Centre for Electron Microscopy Berkeley, USA, at the SuperSTEM in Daresbury, UK and at the Carnegie Mellon University of Pittsburgh in the department of Materials Science and Engineering, to study the grain boundary character distribution (GBCD) of  $\text{Mg}_2\text{SiO}_4$ .

### Dr Alba Matas Adams



Alba joined the Department of Materials as Technical Manager in November 2016. Prior to this, she was a PhD student at ICIQ (Tarragona), researching on new materials for bio- and energy related applications. She is involved in technical and administrative activities for the Centre for Advanced Structural ceramics (CASC) and two projects (XMAT and RESLAG). She has experience working on the development of wide range of materials. She also engages herself in other programmes 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.

## Fellows

<b>Dr Nasrin Al Nasiri</b>	Royal Academy of Engineering Fellow
<b>Dr Samuel Humphry-Baker</b>	Imperial College Research Fellow

## Researchers

<b>Dr Eleonora D'Elia</b>	Research Associate
<b>Dr Daniel Glymond</b>	Research Associate
<b>Dr Dongwoo Kang</b>	Research Associate
<b>Dr Eugenio Zapata-Solvas</b>	Research Associate
<b>Dr Giorgio Sernicola</b>	Research Associate
<b>Dr Iuliia Elizarova</b>	Research Associate
<b>Dr Alba Maria Matas Adams</b>	Research Associate
<b>Dr Oriol Gavalda Diaz</b>	Research Associate
<b>Dr Tommaso Giovannini</b>	Research Associate

## PhD students

<b>Stuart Aberdeen</b>	<b>Alan Leong</b>
<b>Qiaosong Cai</b>	<b>Wirat Lerdprom</b>
<b>Ben Currie</b>	<b>Jack Lyons</b>
<b>Justine Delage</b>	<b>Muhammad Maktari</b>
<b>Max Emmanuel</b>	<b>Annalisa Neri</b>
<b>Claudia Gasparrini</b>	<b>Kristijonas Plausinaitis</b>
<b>Rowan Hedgecock</b>	<b>Jia Hui Teo</b>
<b>Yun-Hao Hsieh</b>	<b>Kathryn Yates</b>
<b>Jindaporn Juthapakdeeprasert</b>	<b>Shitong Zhou</b>

## Visitors

<b>Dr Ozge Akbulut</b>	Sabanci University, Turkey
<b>Dr Moritz Von Witzleben</b>	President of the European Ceramic Society
<b>Dr Qiang Fu</b>	Corning University, USA
<b>Dr. David Noriega, Dr. Laura Megido, Dr. Thi Tan Vu</b>	ArcelorMittal, Spain

## CASC Alumni 2017-2018

<b>Dr Eleonora D’Elia</b>	Teaching Fellow at Imperial College, London, UK
<b>Dr Dimitry Pletser</b>	Waste Management Consultant at Wood Group, Harwell, UK
<b>Dr Kristijonas Plausinaitis</b>	Oracle Consultant at Namos Solutions Ltd, London, UK
<b>Dr Claudia Gasparrini</b>	Research Associate at Imperial College, London, UK.
<b>Dr Annalisa Neri</b>	Principal Scientist at Welland Medical
<b>Dr Eugenio Zapata Solvas</b>	Strategic Research Facilitator (Platforms & Industry) at UCL, London, UK
<b>Dr Daniel Glymond</b>	

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## **RESEARCH**

### **Research Fellow Projects**

❖ **Name: Dr Nasrin Al Nasiri**

Project title: Novel coatings for ceramic gas turbines

Funder: Royal Academy of Engineering

The need to increase the cycle efficiency and reduce noise and NO<sub>x</sub> emissions from engines has promoted the development of ceramic matrix composites (CMC) such as silicon carbide (SiC-SiC). Use of CMCs will lead to a significant improvement in fuel consumption and weight savings of up to 30% compared to Ni-based super alloys. Si-based ceramics have excellent oxidation resistance due to formation of a protective silica layer on reacting with dry air. However, 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 consisting of a minimum of 4 layers requiring a costly application method such as plasma spraying.

The main aim of my work is to develop a reliable single layer of EBC, develop a low cost applying method and studying the corrosion behaviour. I have selected four rare earth monosilicates as promising EBCs based on their thermal

performance: Erbium (Er), Yttrium (Y), Ytterbium (Yb) and Lutetium (Lu). I have developed a patented wet processing technique to apply water-based RE-oxides on CMC samples previously oxidised in dry air to produce protective SiO<sub>2</sub> layer. The samples are subsequently fired to promote reactive sintering and adhesion. The advantages of this non-line of sight method are: 1) it provides better adhesion, 2) it is a low cost and easy to use method, 3) it can be applied to any complex shape and size and 4) it has 100% powder efficiency leading to dramatic cost savings in coating materials. This new coating technology will lead at least to the following advantages: 1- Operational savings (10-15% higher fuel efficiency), 2- Production savings (up to \$100K per engine) and 3-Reduction of emissions by 25-30%. This will result in more efficient, lighter, faster, cheaper, less noisy and less polluting gas turbines.

❖ **Name: Dr Samuel Humphry-Baker**

Project title: Ceramic composites for extreme environments.

Mentor: Dr Luc Vandeperre.

Sponsor: Imperial College Research Fellowship

My research is focused on powder-processing of ceramic composites for extreme environments. This work covers two applications. The first is on highly wear resistant materials used in tools for manufacturing and energy extraction. The second is on materials for nuclear applications. In the latter area, my interests are in materials for high heat-flux reactor components, such as neutron shields and exhaust systems. In both research themes, I study mainly the transition metal carbides and borides. Also common to both is the need to understand and design for harsh conditions such as high temperature, mechanical stress and corrosion.

Part of my research concerns the design of materials with enhanced toughness or damage tolerance from otherwise relatively brittle constituents. Such design principles include combining ceramics with small additions of ductile metallic alloys, alloying multiple ceramics in the form of compositionally complex compounds, and precipitation-strengthening. I process some of these materials using powder consolidation techniques such as vacuum hot-pressing. Others are fabricated with industrial collaborators such as Plansee, Hyperion Materials and Technologies and Tosoh SMD.

Complimentary to this work is my interest in characterising materials in extreme nuclear environments. One such environment is high

radiation fluxes. This work is conducted at UK ion-beam facilities such as the Microscope and Ion-Accelerator for Materials Investigations (MIAMI) and the Dalton Cumbria Facility (DCF). Studies are also being carried out at the Julich Institute's JUDITH facility for plasma-surface interactions. Following irradiations, samples are brought back to college and evaluated using TEM and nanoindentation. The work benefits from on-going collaboration with Tokamak Energy Ltd and their support of a PhD student within the ICO-CDT in Nuclear Energy.

I am also interested in the performance of materials at very high temperatures. A focus of this is deformation studies, which makes use of the vacuum-atmosphere mechanical tester and the high temperature thermal analysis equipment in CASC. The ultimate aim of this work is to map out deformation mechanisms in these materials and thus enable assessment of their service life. A secondary focus of this work is around oxidation at high temperatures. Here I use thermogravimetry to screen composites and their oxidation-resistant coatings, with more systematic studies to understand specific degradation phenomena on industrially-relevant materials. Complimentary tests are also being conducted at high-heat flux testing facilities with external collaborators.

## PDRA Projects

### ❖ Name: Dr Alba Maria Matas Adams

Project title: RESLAG.

Supervisor: Prof Eduardo Saiz.

Sponsor: HORIZON 2020.

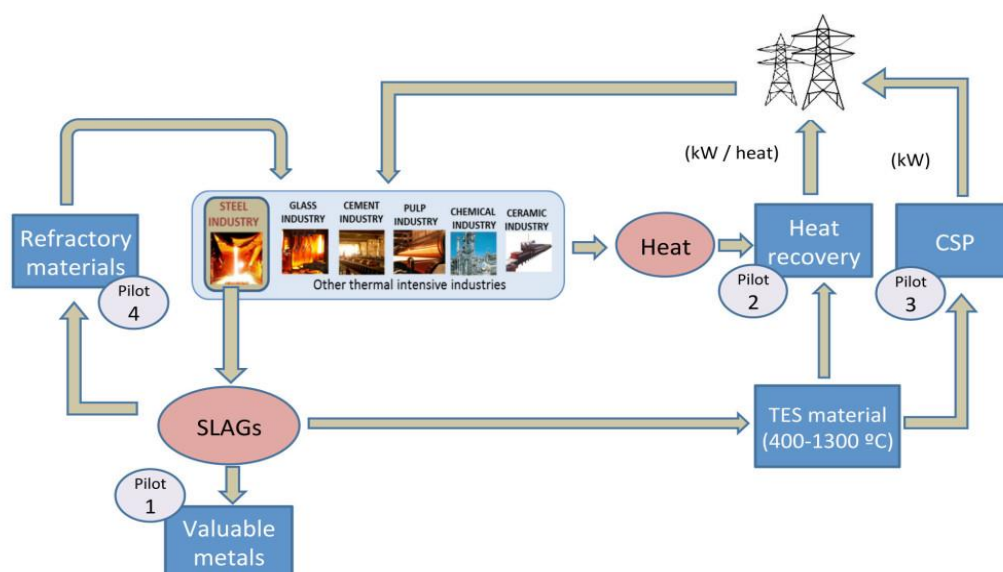
The European steel industry generated about 21.4 million tons of slag in 2012 resulting from steel making. About 24% is not being reused, representing a severe environmental problem in Europe, but also a huge amount of available material for potential recycling. [RESLAG](#) will face this environmental problem by providing 4 eco-innovative industrial alternative applications to valorise the steel slag.

The main objective of RESLAG project is to valorise the steel slag that is currently not being recycled (right now it is partially landfilled and partially stored in the steel factories) and reuse it as a raw

material for 4 innovative applications that contribute to a circular economy in the steel sector with an additional cross-sectorial approach. These applications will be demonstrated at pilot level and led by end-user industries. Altogether open enormously the range of possibilities of taking profit from slag not only for the steel sector but also for many other sectors.

RESLAG is coordinated by CIC Energigune (Spain) and has 19 academic and 4 industrial partners, Imperial College is a fundamental partner involved in the mechanical, thermal and optical characterization of the slag pebbles as receive and the produced refractories used in the steel industry.

**Figure 1 – RESLAG Project Main Concept design.**



❖ **Name: Iuliia Elizarova**

Project title: Manufacture using Advanced Powder Processes (MAPP).

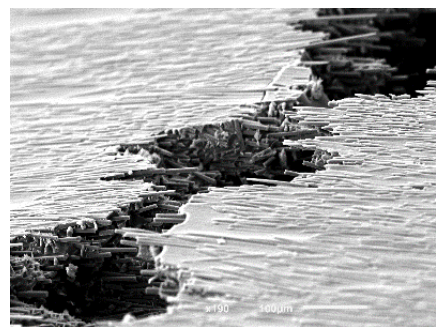
Supervisor: Prof Eduardo Saiz, Dr Luc Vandeperre and Dr Finn Giuliani.

Sponsor: EPSRC

[MAPP](#) is the EPSRC Future Manufacturing Hub in Manufacture using Advanced Powder Processes. MAPP's vision is to deliver on the promise of powder-based manufacturing to provide low energy, low cost, and low waste high value manufacturing routes and products to secure UK manufacturing productivity and growth. MAPP is led by the University of Sheffield and brings together leading research teams from the Universities of Leeds, Manchester and Oxford, and Imperial College London, together with a founding group of 17 industry partners and the UK's High Value Manufacturing Catapult.

Robocasting, or Direct Ink Writing, is an extrusion-based 3D-printing technique that allows for fabrication of structures of various shapes and sizes and can utilize a range of materials – metals, ceramics, polymers. Ceramic composites represent an important class of materials due to their enhanced properties; there is, therefore, an ongoing effort of development of advanced techniques for their manufacturing. In robocasting, ceramic and other powders are made into printing inks

which are then extruded into desired shapes, followed by the thermal treatment to obtain the final composite. If powders are anisotropic (platelets, fibres) the technique provides an additional advantage of particle alignment which leads to improvement of properties depending on the type of material (i.e. toughness, strength and others). The obtained properties are then tested accordingly.



*Figure 2. Fracture of a 3D printed fibre reinforced composite*

❖ **Name: Dr Daniel Glymond**

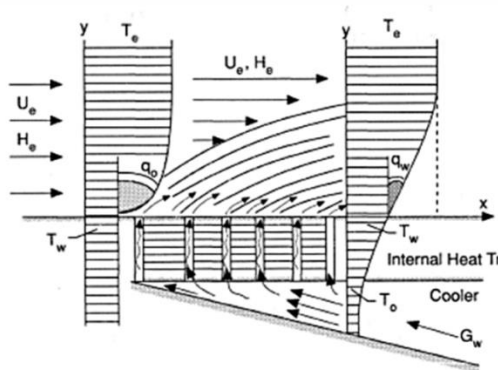
Project title: Transpiration Cooling Systems for Jet Engine Turbines and Hypersonic Flight.

Supervisors: Dr Luc Vandeperre and Prof Bill Lee.

Sponsor: EPSRC.

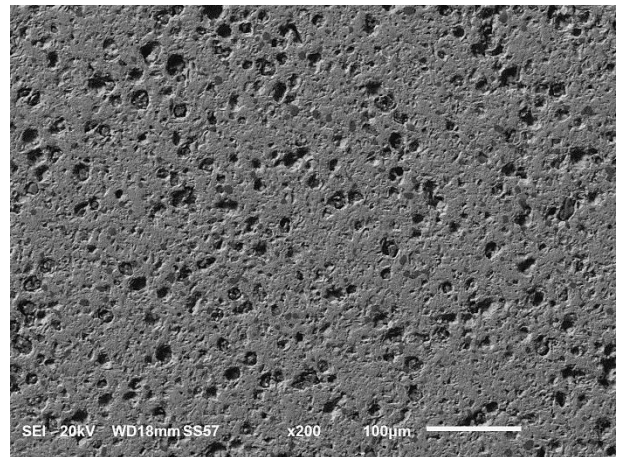
Thermal protection systems for hypersonic flights are expected to have to operate with component temperatures in excess of 2000 °C, leaving only a small group of materials with sufficient high melting points. Ultra-High Temperature Ceramics (UHTC) have become the lead candidates for the development of such protection systems also due to their high

thermal conductivity which enables heat to be conducted laterally thus reducing the maximum temperatures of the sharp leading edge. The basis of transpiration cooling is the introduction of a cool layer of gas between the component and the hot freestream flow, reducing the heat flux to the material (Figure 3).



**Figure 3.** Transpiration cooling system

UHCTs with controlled porosity are suitable for application in transpiration cooling systems. This work addresses the manufacturing of innovative porous UHCTs by establishing a densification route which does not require pressure and allows producing the components in the correct shape without requiring extensive machining or large amounts of material loss. Samples of  $ZrB_2$  with homogenous porous structures were manufactured by partial sintering or by addition of different volume fractions of fugitive inclusions, using starch as a pore former (Figure 4).



**Figure 4.** Pressureless sintered  $ZrB_2$  using starch as a fugitive agent

Characterization of the sintered porous UHCTs was focussed on measuring systematically the relationship between pore structure, porosity and some properties such as thermal conductivity, strength or flow. All this information enables to select the most relevant candidate which is able to maintain excellent thermal and structural properties with increased porosity

Future research will focus on the generation of designed porous structures.

- ❖ **Name: Dr Eugenio Zapata-Solvas**  
Project title: Carbides for Future Fission Environments.  
Supervisor: Prof Bill Lee.  
Sponsor: 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. The synthesis, sintering by hot press and spark

plasma sintering and microstructural characterization by XRD, SEM and TEM of the different Zr-based carbides is done at Imperial College. The research has been focused on the effect of processing and of the impurities in the synthesis of sub-stoichiometric  $\text{ZrC}_{1-x}$ , and  $\text{Zr}_{n+1}\text{AlC}_n$  and  $\text{Zr}_{n+1}\text{SiC}_n$  MAX phases, being able to synthesize  $\text{Zr}_3\text{AlC}_2$  with the highest yield reported till date.

Currently, quinary MAX phases are being manufactured and a study about how the addition of more elements increases MAX phases stability and potentially their yield is under investigation.

➤ **Name: Dr Dongwoo Kang**

Project title: Fabrication of Graphene Coatings on Advanced High Strength Steel

Supervisors: Prof Eduardo Saiz

Sponsor: Arcelor Mittal

Graphene is a 2D material with outstanding mechanical properties, tunable electrical and optical response, chemical versatility, controllable permeability and extremely high specific surface area. It has the potential to revolutionize a wide range of technologies from energy storage devices such as batteries and supercapacitor to membranes. In particular, graphene is a one of the most potential candidate for the in the fabrication of protective coatings and films because its two-dimensional nature and impermeability. The challenge now is how to integrate graphene into

manufacturing technologies for the reliable fabrication of coatings at the large scale.

Due to its relative simplicity and easy technological translation, Electrophoretic Deposition (EPD) is a very attractive coating technology. EPD has been used in the fabrication of thin and thick films from polymers and ceramics. By manipulating the surface charge of the particles, the configuration of the electrodes and the electrical potential or current it is possible to control the characteristics of this coating. Some of the advantages of EPD coatings include improved adherence, homogeneity and density and the possibility of coating complex shapes. There are several reports in the literature on the electrophoretic deposition of graphene coatings on a wide range of substrates. Most of these works use thin flakes obtained through the chemically exfoliated graphene known as graphene oxide (GO). GO is an atomically thin layer of graphene covalently functionalized with oxygen groups that can be prepared by exfoliation of graphite oxide in aqueous solution. It can be produced in relatively larger quantities and if necessary, additional thermal or chemical treatments can be used after deposition for further reduction. The class of materials obtained through the reduction of GO is often described as “chemically modified graphene” (CMG).

In this project we will study the deposition of chemically modified graphene coatings on stainless steel

substrates using electrophoretic deposition. Our focus is on the development of protective coatings and we will study the structure and adhesion of the coatings as a function of the starting material and the deposition conditions. These coatings could be used by themselves or as intermediate films between steel and other protective layers. In collaboration with Acerlor Mittal we will also develop approaches for the scaling up of the coating process such that it can become a feasible route for the coating of steel in practical applications.

➤ **Name: Dr Oriol Gavalda Diaz**

Project title: Ceramic Matrix

Composite Technology Development

Supervisors: Prof Eduardo Saiz

Sponsor: Rolls Royce

The need to increase the cycle efficiency and reduce NO<sub>x</sub> emissions from aero-engines has promoted the development of Ceramic Matrix Composites (CMCs), allowing an increased turbine entry temperature which will lead to a significant improvement in fuel consumption and/or weight savings. Silicon Carbide (SiC) based Ceramic Matrix Composites (CMCs) entered in service in aircraft turbine engines as replacements for some Ni-based superalloys and are currently expected to grow in demand, with the principle CMC developed having a structure formed from SiC fibres, a BN interphase coating and a SiC-based matrix. However, their

stability in the harsh operation environments that form in the hot part of an aero-engine is still a matter of concern.

Hence, in this project we focus on understanding how SiC/BN/SiC CMCs are affected by the stress-environmental solicitations present in the hot part of an aero-engines by understanding the mechanisms of degradation and failure. This study will lead to a better prediction of the lifetime of critical aero-engine components.

➤ **Name: Dr Tommaso Giovannini**

Project title: Porous UHTC's for transpiration cooling of hypersonic flight component

Supervisors: Prof Luc Vandeperre

Sponsor: EPSRC

Lowering the cost of space travel is one of the main challenges faced by the aerospace industry today. Success in this endeavour is contingent on the development of reusable spacecraft construction materials which can perform several missions prior to replacement. This design approach is particularly difficult for leading edge heat shield materials due to the high temperatures and corrosive environments present upon re-entry into the earth's atmosphere. Conventional heat shield materials are either ablative or thermally soaking. Ablative materials absorb heat by vaporizing during re-entry whilst thermal soaks are thermally insulating meaning they absorb heat

and radiate it away from the spacecraft. Both these types of materials are single use because of the significant damage they incur during re-entry. The key to making these components reusable may lie in a process known as transpiration cooling in which gas from an internal reservoir is bled through the heat shield. This operation serves two distinct purposes. The first is the removal of heat from the spacecraft via convection cooling. The second is the formation of a thin protective gas layer on the craft's outer surface which protects it from corrosion. My research focuses on porous ZrB<sub>2</sub> components used for these applications and involves their processing and microstructural characterization.

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## PhD Projects

➤ **Name: Cyril Besnard**

Project title: Si Doping of Boron Carbide.

Supervisor: Dr Luc Vandeperre and Dr Finn Giuliani.

Sponsor: DSTL.

The aim of this project, supported by the Defence Science and Technology Laboratory of the UK, is to develop novel ceramics for use in armour. Lightweight impact resistance ceramics are still under development.  $B_4C$  is attractive and has already been used for this application for many decades. However catastrophic failure occurs in  $B_4C$  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  $B_4C$ . Therefore the aim of this project is to produce meaningful quantities of Si doped  $B_4C$  which can be used for high speed impact testing. This project is also in collaboration with the shock physics group at Imperial College.

➤ **Name: Qiaosong Cai**

Project title: Robocasting of complex structural ceramics.

Supervisor: Prof Eduardo Saiz, Dr Luc Vandeperre and Dr Finn Giuliani.

Sponsor: CASC Industrial Consortium.

Robocasting is a distinct additive manufacturing technique that can be used to print complex structural ceramics. In robocasting, inks are extruded out through a nozzle to build 3D objects layer by layer. By using colloidal inks, hydrogel inks, emulsion-based inks or foam gel inks, dense or porous ceramics can be printed by robocasting.

With the aim to expand the applications of structural ceramics, a novel complex structure which is called core structure is designed.

This structure is achieved by co-extrusion of two different kinds of inks. One of the concepts is printing electronic conductive metal fibres shielded with dense ceramic shells. Another concept is producing filaments with porous centre and dense shell with a potential application of heat exchange. The inks for dense shells and metal fibres are prepared by using Pluronic solutions as the particle carriers. This kind of hydrogel ink is sensitive to the temperature and has a suitable rheology for robocasting. The porous inks used are emulsion-based inks in which the macroscopic shape and microscopic porosity of the objects can be controlled easily.

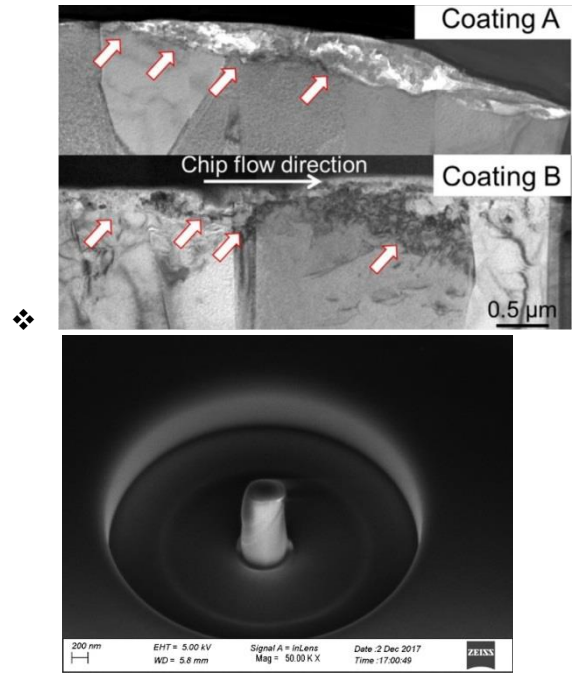
❖ **Name: Tommaso Giovannini**

Project title: Plastic behaviour of ceramics coatings – an experimental & computational characterization.

Supervisors: Dr Finn Giuliani and Dr Daniel Balint.

Sponsor: SECO Tools.

With the global cutting tool market predicted to reach \$54.6 billion by the year 2021, there is a strong need for improved cutting tools which are able to withstand the stringent time and cost requirements of modern manufacturing. Thin ceramic coatings ( $\leq 10 \mu\text{m}$ ), deposited onto cemented carbide inserts using chemical vapour deposition (CVD), have been used since the end of the 60s to improve cutting tool performance and lifetime. Although the quality of the deposited coatings has improved considerably a deeper understanding of the microstructural mechanisms which govern wear in the coatings is required to allow for further improvement of coating performance. This understanding is closely linked to the localized plastic deformation behaviour which has been observed in coatings after metal cutting operations (**Figure a**).



**Figure 5a)**-Bright field TEM images in  $\alpha\text{-Al}_2\text{O}_3$  coatings highlight the presence of dislocation activity close to the surface ( $0.5 - 1 \mu\text{m}$  deep)<sup>1</sup>. **b)** Micropillar compression performed on the  $\alpha\text{-Al}_2\text{O}_3$  coatings highlights plastic slip activity in the coating materials at reduced length scales.

High temperature nanoindentation and micropillar compression experiments (**Figure b**) can be used to provide an initial characterization of the coating's plastic response at increasing temperatures. Crucially, these experiments can also be used to extract inputs for a discrete dislocation modelling (DD) framework aimed at better understanding the plastic behaviour of the coatings. By investigating the relationships existing between plastic activity, the presence of defects, such as grain boundaries and thermal cracks, and overall cutting tool performance, the hope is to highlight which microstructural features are associated with localized coating failures. These are closely linked to tool lifetime and their elimination presents a strong opportunity for

improving overall cutting tool performance.

Reference:

1. *Microstructure and wear mechanisms of texture-controlled CVD  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> coatings*. R. M'Saoubi, O. Alm, J.M. Andersson, H. Engstrom, T. Larsson, M. P. Johansson-Joesaar, M. Schwind. *Wear*, 376-377, p:1766-1778, 2017.

➤ **Name: Yun-Hao Hsieh**

Project title: Ceramic Wasteforms for Advanced Fuel Cycle Reprocessing.

Supervisors: Prof Bill Lee and Prof T.D. Waite (UNSW, Sydney, Australia).

Sponsor: The UNSW Tyree Scholarship.

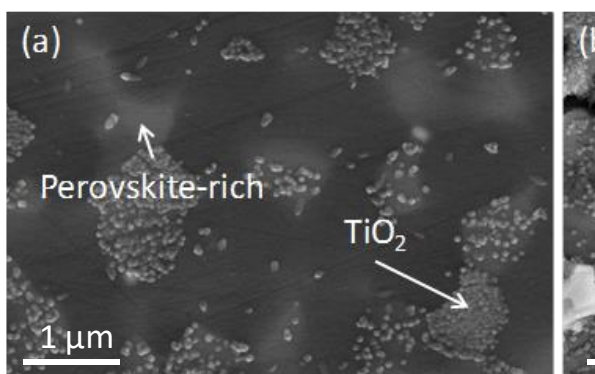
Many advanced reprocessing technologies have been designed and developed to meet the future nuclear waste policies and further separate waste. One of the most promising advanced reprocessing methods, EURO-GANEX (Group Actinides Extraction), is nowadays being developed and aim to further separate both major (U, Pu) and minor (Np, Am, and Cm) actinides together from fission products. In that context, new advanced immobilization matrices capable of accommodating such modified waste streams have to be investigated<sup>1, 2</sup>.

Synroc and borosilicate glass are potential wasteforms that

have proven themselves to be efficient to immobilize high-level nuclear wastes (HLW)<sup>3</sup>. In this project, a new composition of Synroc, "Synroc-Z", is designed and characterized. The primary modification is to reduce the amount of zirconolite phase, which acts as the main host for actinides.

Synroc-Z has been prepared via hot uniaxial pressing (HUP), with optimized process conditions to obtain dense samples. A well-known borosilicate glass, R7T7, was melt to compare with HUPed Synroc-Z. In both wasteforms, their durability via the MCC-1 test at 90°C and microstructure after corrosion was tested.

The mass loss before and after leaching test for Synroc-Z was negligible (< 0.02%), while for the R7T7 glass it was relatively larger (>0.35%). Nano TiO<sub>2</sub> particles (~50 nm) were formed on the perovskite-rich surface. The thickness of TiO<sub>2</sub> skin was determined via AFM which was ~60 nm. In the leached sample, which were polished to 1  $\mu$ m, it can be clearly observed that TiO<sub>2</sub> particles were assembled on the brighter contrast area while the TiO<sub>2</sub> particles were dispersed on the rougher (15  $\mu$ m polishing) Synroc-Z surface but dense on the edge of holes. The leached R7T7 glass formed Al, Zn and Si rich layer on the surface. The ratio of Al, Zn and Si of layer on the surface is ~1: 2: 4 while the composition of unleached R7T7 glass is ~2: 1: 18.



**Figure 6.** SEM images of 20 wt.% waste loading HUPed Synroc-Z samples with (a) 1  $\mu\text{m}$  and (b) 15 mm polishing finish. The  $\text{TiO}_2$  particles compact on perovskite-rich regions. Samples with 15  $\mu\text{m}$  polished shows dispersed  $\text{TiO}_2$  skin but dense in some edges of holes.

#### References:

1. *Advanced Separation Techniques for Nuclear Fuel Reprocessing and Radioactive Waste Treatment* K. L. Nash and G. J. Lumetta, . Elsevier Science, (2011).
2. *Development of a New Flowsheet for Co-Separating the Transuranic Actinides: The "EURO-GANEX" Process.* M. Carrott, K. Bell, J. Brown, A. Geist, C. Gregson, X. Hères, C. Maher, R. Malmbeck, C. Mason, G. Modolo, U. Müllich, M. Sarsfield, A. Wilden, R. Taylor. *Solvent Extraction and Ion Exchange*, 32[5], p: 447-67, 2014.
3. *Radioactive waste forms for the future.* W. Lutze and R. C. Ewing. North-Holland, 1988.

- **Name:** Jindaporn Juthapakdeeprasert  
**Project title:** Development of Multifunctional Cement Kiln Refractory Coatings.  
**Supervisor:** Prof Bill Lee.

Sponsor: SCG Cement-Building Materials.

For decades, the cement industry has been consuming numerous of fuel to generate heat in producing cement. Approximately 10-15% of the heat generated from these fuels are lost to the atmosphere through refractories and kiln external surface. The heat loss in the cement production can be reduced by developing a coating with a property of high thermal emissivity. This research is focusing on developing a high emissivity coating for refractory used in cement production. Cerium oxide is use as emissive materials. Di-alumina phosphate is used as a binder. The reactions between cerium oxide and di-alumina phosphate is studies. The Emissivity is analysed with apparatus emissometre respectively. Microstructure, phases analysis along with other properties such as its chemical resistance and thermal shock resistance will also be investigated to ensure the coating will give excellent performance when is used in the cement production environment.

#### ❖ **Name: Alan Leong**

**Project title:** Graphene Coatings for Pipelines.

**Supervisors:** Prof Eduardo Saiz and Dr 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.

➤ **Name: Wirat Lerdprom**

Project title: Impact of Fast Firing on Phase Evolution in White-ware Ceramics.

Supervisor: Prof Bill Lee.

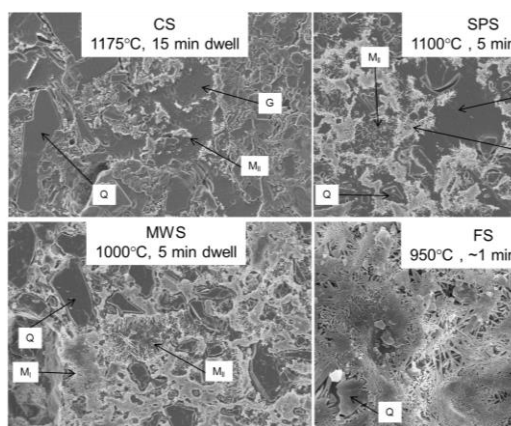
Sponsor: Cementhai Ceramics Co., Ltd, Thailand

Porcelains are clay based materials, used extensively in household, scientific and engineering applications, which are produced via viscous flow sintering. The sintering process of porcelains is not only to densify the green body, but also to induce mineralogical phase changes. Densification and phase evolution are influenced by sintering conditions i.e. heating rate, dwell time, atmosphere, and temperature.

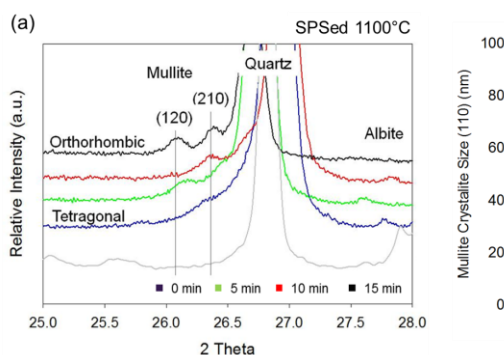
New advanced sintering processes have been introduced aiming to improve product quality and energy usage efficiency such as conventional fast firing (CS), spark plasma sintering (SPS), flash sintering (FS) and microwave sintering (MWS), each of which has different process parameters.

The aim of this work is to investigate microstructural and physico-mechanical property changes (apparent bulk density, water absorption, Vickers hardness, and fracture toughness) in a porcelain body sintered using the 4 techniques and consider energy, financial and other aspects to determine which is most likely to find industrial application. The study involves investigation of mullitization, glass formation, and

quartz dissolution from the different sintering techniques using X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and energy-dispersive spectroscopy (EDS). Finally, the ultimate goal of this work is to develop understanding of process parameters i.e. heating rate, pressure, atmosphere, electric field, and microwave radiation on mineralogical composition and densification in porcelains.



**Figure 7.** SEM-SE images of conventional sintered (CS), spark plasma sintering (SPS), microwave sintering (MWS), and flash sintered (FS) porcelain samples; showing etched samples using 20% HF (Ml=primary mullite, MII=secondary mullite, G=glass, and Q=Quartz).



**Figure 8.** (a) XRD patterns of the SPSed samples showing different mullite crystal structure as a function of dwell times, (b) mullite crystallite size and aspect ratio of

*the porcelains sintered using different techniques.*

❖ **Name: Annalisa Neri**

Project title: Development of a Novel Wound Management Dressings.

Supervisor: Prof. Eduardo Saiz.

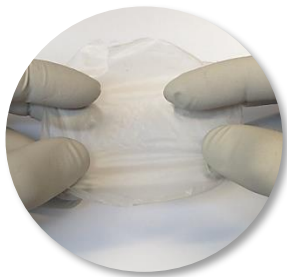
Sponsor: Welland Medical Ltd.

Wound management represents a challenging field, not only in research and product development but also in clinic, where patients present an array of wound types. In particular, infection is a common problem in wound healing: this can result in reduced healing rates, prolonged hospitalization time and increased care costs.

The focus of this study is to develop and characterise a novel dressing comprising collagen and honey which is able to actively promote healing while preventing infection in a variety of wound types.

Collagen presents several properties that are desirable for a wound dressing: strong biocompatibility, weak antigenicity, biodegradability and in addition it can terminate the chronic state of a wound. The honey component exhibits anti-inflammatory and anti-bacterial properties, while also allowing for manipulation of the wound pH. Herein the combination of collagen and honey is investigated for the design of a novel bioactive wound dressing film able to actively promote healing. This material is intended to

adhere and conform to the wound site and to degrade in contact with wound exudate. The collagen-honey films produced (Fig. 9) are being characterised in terms of chemical homogeneity, degradation rate, mechanical properties, antibacterial activity and cells response.



*Figure 9. Collagen-honey based films*

- **Name: Kristijonas Plausinaitis**  
 Project title: Adsorption of Heavy Metals and Radionuclides on Cement Phases.  
 Supervisors: Dr Luc Vandeperre and Prof Mary Ryan.  
 Sponsor: Amec Foster Wheeler.

Lead (Pb) is one of the constituents in intermediate level radioactive waste. Due to its high toxicity, it is of particular importance to understand the behaviour of soluble Pb ions, in particular their sorption on and migration within the cement backfill, one of the main proposed engineering barriers for geological waste disposal.

Of particular interest for understanding the long term safety of these engineered barriers is how the evolving cement matrix may affect efficacy of capture of mobile

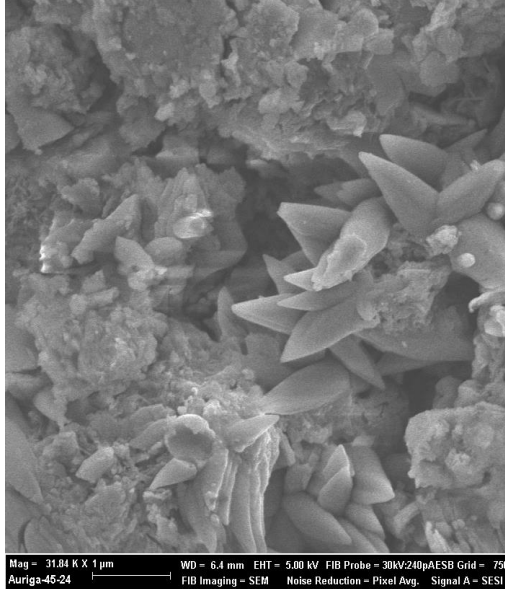
species by sorption and /or precipitation processes.

In this work, batch sorption analysis of Pb on to hydrated Nirex Vault Reference Backfill (NVRB) cement powder indicates a Langmuir like process: a linear uptake followed by a plateau due to saturation at increasing concentrations. Leaching experiments were also carried out and suggested that the sorption process is reversible.

The homogeneity of surface interaction was analysed using spatially resolved chemical analysis. In particular we have used scanning electron microscopy (SEM), energy dispersive X – ray spectroscopy (EDS) and secondary ion mass spectrometry (SIMS) to provide information on surface concentrations. These data indicated that the uptake of Pb is relatively homogenous across the cement surface with no sites with much higher concentration observed. This suggests that lead is primarily uptaken into the calcium silicate hydrate (C – S – H) gel since this is the most abundant phase in the cement. In addition localized and unique crystal clusters were also observed on samples exposed to higher concentrations of Pb in solution.

The results indicate that a number of previous studies where the Pb was introduced during cement hydration and where Pb was found not to be released significantly are misleading from a

geological waste repository perspective where the Pb will only come in contact with the cement after hydration is completed.



**Figure 10.** SEM figure displaying unique crystal cluster formed on the NVRB cement sample exposed to Pb.

#### ❖ Name: Giorgio Sernicola

Project title: Developing small scale fracture tests for polycrystalline diamond.

Supervisors: Dr Finn Giuliani and Dr T. Ben Britton.

Sponsor: Element Six.

Polycrystalline diamond composites (PCD) were first sintered in the 1970s following research efforts focused on producing new, more durable materials to use as cutting tools. These composites are characterised by a complex microstructure composed of two stark different phases, hard and brittle diamond grains and a network of softer and ductile cobalt. Given the high

volume fraction of brittle phase, life of these tools is dominated by their fracture behaviour and catastrophic failures that still represent the major issue for their application.

In the last three decades, improvements of the fracture properties of brittle materials have been sought through the development of new insights on toughening mechanisms, typically involving microstructure control that focuses on crack deflection at grain boundaries and interfaces. However, these are often difficult to engineer, as changing microstructural processing (e.g. through heat treatment, chemistry or powder processing) does not result in a one-to-one correlation with performance, since the influence of microstructure on crack path is varied and complex. Recent developments on characterisation at the micro-scale therefore present an opportunity to broaden our understanding of the role of individual factors on the bulk performance.

To investigate the fracture properties of individual features (i.e. individual crystallographic planes, grain boundaries or interfaces), we developed an innovative testing method. This approach is based on the double cantilever wedging to measure the fracture energy evolution with crack during stable growth and was successfully applied at the micron scale inside a SEM. Direct view of the crack growth in our sample and measurement of the energy

absorbed during fracture, without use of load-displacement data, is afforded through the combination of a stable test geometry with an image based analysis strategy.

In addition to these tests, we have targeted characterisation at the role of microstructure on crack paths in polycrystalline diamond. Our focus has been on using high angular resolution EBSD combined with microindentation, to correlate intra-granular residual stresses gradients, due to thermal expansion mismatches, to crack deflection. It was found that the crack can follow the grain boundaries if grains are small but tends to deviate along (111) in coarse grains, yet stress gradients disrupt homogeneity of individual grains and are able to deflect the crack.

Exploitation of these novel techniques allows us to gather new insights on the mechanical properties of advanced ceramics that can usher in a new way of engineering the microstructure to obtain tougher ceramics.

➤ **Name: Jia Hui Teo**

Project title: Designing Ceramic Matrix Composites for Ceramic Armour.

Supervisors: Dr Luc Vandeperre and Prof Eduardo Saiz.

Sponsor: DSO National Laboratories.

Ceramics are hard materials, which makes them ideal for armour but their brittleness and weakness in

tension is detrimental to performance. It is generally understood that impedance mismatches are bad for any armour systems as they are points of reflection, generating tensile stress waves which should be avoided. In this work, varying microstructures will be created using materials with different impedances to understand the reflection of waves at such interfaces on the microstructural level. Consequently, a better understanding of how the microstructures can be tailored to create materials that are able to reflect compressive waves progressively across the entire thickness instead of generating a large tensile wave at the rear end of the ceramic which will likely cause the ceramic to shatter. Delaying the failure of the ceramic gives it more time to defeat the ceramic, allowing sustained projectile erosion which could help improve the ballistic performance of the ceramic.

➤ **Name: Kathryn Yates**

Project title: Investigation of helium bubble behaviour in a lattice-damaged FCC metal

Supervisors: Dr Finn Giuliani

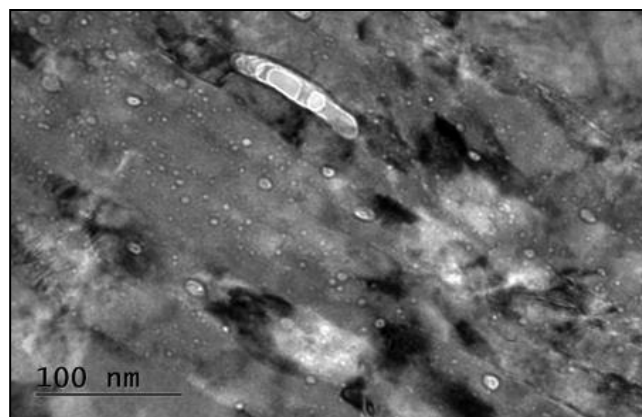
Sponsor: AWE

Further knowledge of helium bubble formation and mobility within fcc metals is required to understand and predict the properties of plutonium and its alloys for safe-handling, use, and long-term storage. Self-irradiation within plutonium metal results in

lattice damage, compositional changes, and helium accumulation at a rate of 41 appm/year due to  $\alpha$ -decay. These phenomena are thought to affect the materials mechanical properties and corrosion behaviour.

In the absence of  $\alpha$ -emitting material, surrogate metals with the same crystal structure (fcc) for example palladium can be subjected to radiation effects such as helium ion implantation and heavy ion bombardment.

Samples of palladium are irradiated using the 5 MV Tandem Pelletron ion accelerator at the Dalton Cumbria Facility (DCF). Helium is implanted to a range of doses to simulate different ages of plutonium, and lattice damage will be created by heavy ion bombardment. Analytical techniques include TEM imaging to determine bubble sizes, and distributions, EELS for bubble atom densities, in-situ heating of TEM samples to observe bubble mobility and growth, and indentation testing to understand effects of He and damage on the materials' mechanical properties.



**Figure 11.** TEM image taken using a JEOL 2100F at 200 kV to show (left) Palladium irradiated with  $3.5 \times 10^{17}$  He ions/cm<sup>2</sup> using the 5 MV Tandem accelerator at the DCF. Bubbles can be seen across the grains. (right) Palladium irradiated with Pd ions to a level of 0.234 dpa, using the 5 MV Tandem accelerator at the DCF. Dislocation loops and damage features can be seen.

➤ **Name: Justine Delage**

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

Supervisors: Dr Nasrin Al Nasiri and Dr Eduardo Saiz

Sponsor: Rolls Royce

Since the 1980s, fibre-reinforced ceramic matrix composites (CMCs) are the subject of extensive developments as candidate materials for structural applications in extreme environments, due to their excellent thermomechanical properties. A promising use of SiC/SiC CMCs would be the replacement of metallic super alloy components of aircraft engines. CMCs are light weight and can be used at higher operating temperatures than metallic

components. Thereby, their uses will increase the efficiency of the engine and reduce the consumption of fuel, leading to less pollution and economic gains.

Nonetheless, the main complication with CMCs is that they exhibit a complex fibre related crack propagation during fracture which makes their mechanical behaviour hard to fully understand and to model. More specifically, the measurement of the fracture toughness, which describes the resistance to crack propagation of a material, is particularly complicated because of the composite fracture mechanisms. These multiple mechanisms don't lead to a single straight crack initiating at the notch tip but to jagged macroscopic crack due to a combination of matrix microcracks, fibres bridging and fibres pullout mechanisms.

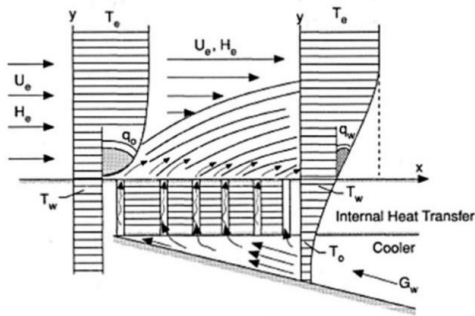
The present study focusses on the understanding of the fracture behaviour, and especially on measuring the fracture toughness of SiC/SiC CMC. To do that, mechanical tests have been carried out such as tensile tests, in-situ and ex-situ bending tests, and in-situ double cantilever beam tests. Tensile tests will determine the resistance to crack propagation by notch sensitivity testing at ambient temperature. In-situ bending tests will allow to observe in-situ the development of cracks within the composite microstructure and to identify and understand better the fracture mechanisms of the

composite. Ex-situ bending tests, on the other hand, will study the fracture behaviour of the composite by means of the evaluation of resistance curve (R-curve). Finally, in-situ double cantilever beam will measure the energy release rate  $G$  to evaluate the interlaminar fracture behaviour. The comparison of these tests and the work of fracture lead to a better understanding of the fracture behaviour of this composite and to learn how to design with them for engineering applications.

➤ **Name: Rowan Hedgecock**

Project title: Design of Porous Materials for Transpiration Cooling  
Supervisors: Prof Luc Vandeperre  
Sponsor: EPSRC

Thermal protection systems for hypersonic flights are expected to have to operate with component temperatures in excess of 2000 °C, leaving only a small group of materials with sufficient high melting points. Ultra-High Temperature Ceramics (UHTC) have become the lead candidates for the development of such protection systems also due to their high thermal conductivity which enables heat to be conducted laterally thus reducing the maximum temperatures of the sharp leading edge. The basis of transpiration cooling is the introduction of a cool layer of gas between the component and the hot freestream flow, reducing the heat flux to the material (Figure 12).



**Figure 12** *Transpiration Cooling System*  
Studies into the use of porous ZrB-2 have shown partially hot pressed ZrB-2 to have sufficient fluid permeability to be effective for transpiration cooling. The current work focuses on control and characterisation of porosity and pore structure during partial hot pressing. Testing will involve ensuring gas flow through the material is homogenous and that the material will not densify further at temperature expected to be seen during operation.

Future work will be to develop a dense-channelled substructure to carry the porous skin and deliver the transpired fluid.

➤ **Name: Max Emmanuel**

Project title: High temperature deformation and in situ fracture measurements to understand grain size/binder effects in hardmetals  
Supervisors: Dr. Finn Giuliani and Dr. Ben Britton  
Sponsor: SECO

The study, at this phase is concerned with examining the role of microstructure and binder chemistry on interface properties in regard to crack growth. Different types of WC/WC grain boundaries

(in terms of CSL) making up the material have been identified. Work is being done to fabricate DCBs around these boundaries with the aim of understanding interface properties. A WC-10wt%Co sample has been used so far. Future work would be concerned with studying cemented carbide samples with large grains ( $\sim 10\mu\text{m}$ ) of 6wt% cobalt content and chromium additions. Chromium is added to inhibit grain growth. Interfaces containing chromium are to be studied at this point while a reference material lacking the inhibitor is to be used as a standard.

➤ **Name: Jack Lyons**

Project title: Development of MAX phases for nuclear cladding  
Supervisors: Dr. Finn Giuliani  
Sponsor: EPSRC

The aim of the project is the development of Zr-based MAX phases for fuel cladding in fission reactors of future nuclear power plants. The project is split into two parts; firstly, developing a fundamental understanding of the mechanism of formation of the MAX phases. This will allow for greater ability to manipulate the properties and formula of MAX phases for specific applications. Secondly investigating the inhomogeneous elastic properties within the MAX phase through the use of Raman spectroscopy.

➤ **Name: Benjamin Currie**

Project title: Study into the Suitability of WC as a Neutron Shielding Material Within Future Fusion Reactors

Supervisors: Dr Samuel Humphry-Baker and Prof Luc Vandeperre

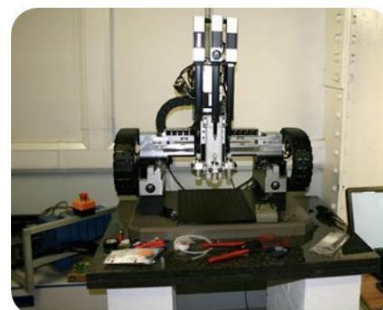
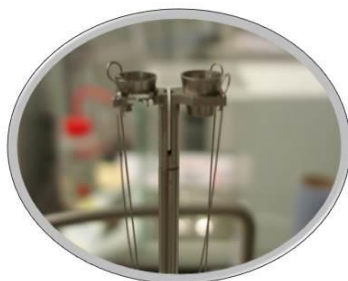
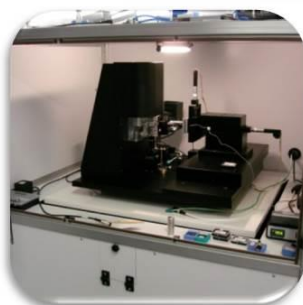
Sponsor: Tokamak Energy and EPSRC

Compact spherical tokamaks will require extremely effective neutron shielding materials in order to ensure that the huge fluxes of highly energetic neutrons produced within do not cause significant heating or damage to more critical components. This becomes crucial when considering the superconducting magnets used to confine the plasma. Only a small amount of heating in these components will lead to significant losses in the efficiency of the reactor. The challenge of finding a suitable material becomes even more difficult when geometry is taken into consideration. The width of the central column defines the geometry of the rest of the reactor. It is therefore very important that this width is kept to a minimum, including the width of the shielding of the central column.

Tungsten carbide (WC) is an extremely effective attenuator of neutrons and gamma rays. It has also been used extensively in the tooling industry, allowing companies like Tokamak Energy to use existing supply chains. For these reasons, WC shows promise and has been selected as a candidate

material for neutron shielding in some of the fusion reactors of the near future. Although WC does show promise, its properties within the fusion environment are still little understood. This project aims to use techniques such as nanoindentation, mutual indentation, compressive creep testing, SEM and TEM on samples at differing temperatures and with differing levels of ion induced damage to gain a more thorough understanding of how the properties of WC might evolve during reactor operation.

## CAPABILITIES AND FACILITIES

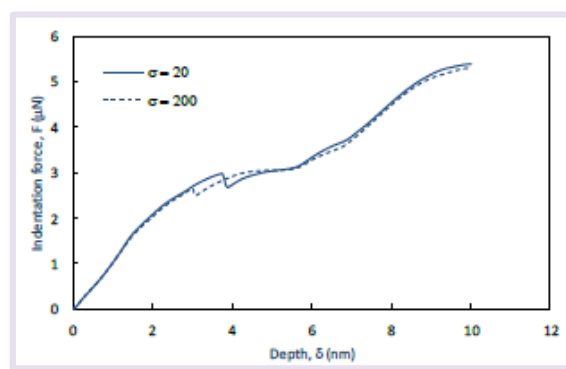


Although the purchasing and installation of large pieces of equipment by the Centre using the funding from the original CASC project is now completed, we continue to improve our experimental capability in this area using funds from other sources.

All equipment is available to the UK ceramics community. Here you will find a list of some of the equipment that we have, and if you wish to use any of these facilities, or have any question, please contact Garry Stakalls ([g.stakalls@imperial.ac.uk](mailto:g.stakalls@imperial.ac.uk), 020 7594 6770).

### **Nano-indenter**

The high temperature nano-indenter, manufactured by Micro Materials, is located in the Structural Ceramics laboratory, on the basement of the Royal School of Mines (RSM), taking advantage of the better control of air, temperature and the reduced vibration levels. As well as being fully instrumented, the nano-indenter operates at temperatures up to 750°C.

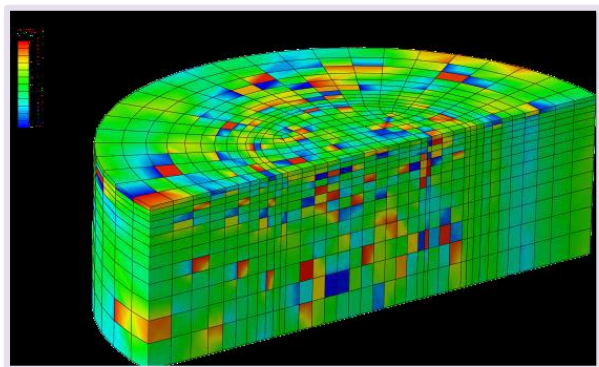


Usage of the nano-indenter is high, and results obtained have been reported at international meetings including the Fall MRS conference (Boston), the American Ceramics Society meeting (Daytona Beach) and at the ICMCTF (San Diego).



*Nanoindenter available at CASC*

## Server



CASC's multiprocessor server allows to solve complex and node rich finite element simulations, such as a crystal plasticity simulation including soft and hard slip systems in MgO. Three dimensional crystal plasticity simulations are being carried out using parallel processing on the CASC cluster. It has been used to simulate the relation between primary and secondary slip systems activation and hysteresis, and the

softening observed in the indentation force displacement response. A normal random distribution of the critical resolved shear stress results in a lower drop in the indentation force. This was 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.

## Freezer 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 of eliminating solvents by keeping the material structure intact for further processing, like for example sintering.

We currently use this process for drying freeze-cast materials like alumina, zirconia, zeolites and graphene oxide.



*Freeze dryer at CASC laboratory*

## Thermodynamic software

We purchased the FactSage version 6.1 from GIT Technologies, together with three substance databases.

A multi-user license for phase equilibria software has also been purchased from the American Society. This thermodynamic calculation software is available over the network to anyone in the CASC offices and has been applied to a range of projects including Si-stabilised B<sub>4</sub>C and high temperature annealing of TiAlN, thermal treatments of high alumina castable refractories and producing composites of B<sub>4</sub>C and SiC.

## Thermal analysis

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

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

Netzsch have provided multiple training sessions, and all three items of the equipment are up and running. The facility is heavily used and has a high usage by external users.

The **dilatometer** has two set-ups:

1. An alumina tube and pushrod for measurements up to 1600°C.
2. A graphite set-up for measurements up to 2400°C.



Crucibles of the high temperature combined TGA DTA

In-house developments in the past year have made it possible to use the dilatometer for hardness measurements and even creep measurements test have been done. Examples of CASC projects using the dilatometer are the measurement of the thermal expansion of refractory materials to estimate 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  $\text{ZrB}_2$  and  $\text{Al}_2\text{O}_3$  and the analysis of cracking due to shrinkage in geopolymers and sintering of silicon carbide-boron carbide composites.



The **combined TGA-DTA** has been used to quantify mass loss during drying of geopolymers, to analyse the decomposition of magnesium phosphate and magnesium silicate cements for nuclear waste treatment, to study silicon carbide or mullite sintering, to perform analysis of UHTC oxidation, to determine carbon yield from various ceramic additives and for characterisation of raw materials in general. Usage for third parties included work with Professor Jon Binner (Birmingham University), Loughborough University, and Dr Bai Cui (University of Illinois), as well as characterisation of derivative products from commercial paper mills and work with Morgan Technical Ceramics.

The equipment to measure **thermal diffusivity via laser flash** has been extensively used 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 Mike Reece at Queen Mary College (thermo-electric materials).

An additional set up has been installed with the TG/DTA analyser to measure the specific heat capacity. This year we improved the range of the measurement up to 1650 °C thanks to a high accuracy rhodium furnace.

### 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 second frame has induction heating up to 1200°C.

The equipment is used in work with diverse industrial partners such as Seco Tools AB (Sweden) and for projects like “Turning waste from steel industry into valuable low cost feedstock for energy intensive industry” (RESLAG). 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 UHTCS’s and commercial refractories.

## Vacuum hot press

The vacuum hot press from FCT Systems is fully operational.



The press operates at temperatures up to 2400°C for sintering and 2100°C for hot pressing with a maximum force of 250 KN, and can be used at atmospheric pressure or under vacuum. Large samples can be fabricated, as dies with diameters as large as 8 cm can be used.

Its use in CASC projects includes the preparation of a wide range of materials such as silicon carbide, boron carbide and composites, aluminium nitride alloys, zirconium carbide, tantalum and hafnium carbide, joining of UHTC's, glass ceramic-SiC composites, ultra-light SiC structures and mullite.

Its 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.

It is also used by other university groups to perform tests on forging of functional ceramics for Professor Alford (Imperial College London) and for treatment of UHTC precursors for Professor Binner (Loughborough University).

## Vacuum furnace

The vacuum furnace can be used to heat up a volume of 5 cm in diameter and 15 cm tall to temperatures up to 2500°C, under vacuum or under a mixture of gasses.

Opposed viewing ports allow observation of the sample during heating, and a sample elevator and cooling chamber allows for fast quenching. The equipment has been used in the sintering of ceramics and metal-ceramic composites as well as for the analysis of glass and metal wetting on ceramic substrates.

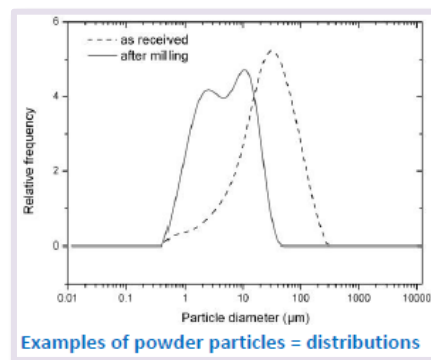
## Wet grinding mills

We 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 of Materials provided funds to acquire a laser particle size analyser. The equipment is able to determine size distribution using scattering of light by particles in dilute solutions and has the ability to measure particles with diameters ranging from  $10^{-2}$  to  $10^{-4}$   $\mu\text{m}$  without changing any optics.



## High Temperature elastic properties by impulse excitation



In 2013 we installed a piece of equipment to determine the Young and shear modulus as well as the Poisson's ratio for different materials. The measurement principle is based on the relationship between shape, density and stiffness and the natural vibration frequencies of a sample. For example, to determine 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  $1750^{\circ}\text{C}$  in air or inert atmosphere. Hardware and software enables fully automated excitation and measurement, making it possible to investigate the variation of the elastic properties with 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.

## 3D Printer

Another important piece of equipment at CASC is a robotic assisted deposition system from 3D Inks (USA). This system can print 3D structures using continuous extrusion. The movement of the printing head can be controlled, with submicron precision. The printer allows the combination of three different inks to fabricate multiphase structures.



In addition to this one, this year, a new 3D printer was purchased, a Micro Plus 3D printer from EnvisionTEC. This piece of equipment can produce functional parts with exceptional surface quality without sacrificing speed. The materials available for the Micro Plus line cover a wide range of applications, including jewellery, toy, medical, industrial design, engineering, and more.



### Laboratory Mixing Extruder (LME)

The Dynisco Polymer Test LME Laboratory Mixing Extruder can be used to evaluate the processability of a variety of plastics and rubbers prior to production. From very fine powders to coarse materials, the LME will meet many extruding needs. It possesses a moveable header and dial gage that allows for constant mixer adjustability and allows for various extrudate mix levels in a single sample run. It can be used in the production of polymer blends or alloys. Mixing may be independently adjusted such that agglomerates of additives, such as fillers or pigments, may be accurately controlled.



It is a three-part system: Extruder with Take Up, and Chopper Accessories. Maximum temperature 400°C and Variable speed control, 5 to 260rpm.

### 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 was also installed in 2013. The microscope has a modular design that facilitates the installation for different set-ups to allow *in-situ* experiments like mechanical testing or freezing of colloids.

### **Rapid prototype (CNC) milling machine**

Rapid prototyping is the dramatically transformation of a design and manufacturing processes of a physical part. This milling machine has answered the call for a cost-effective, high precision and compact solution.

It is used to create realistic models, functional prototypes and moulds and is compatible with a wide range of materials. It is able to produce 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 orienting” 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) was installed at CASC in 2015.

This includes also the Carbon-Sulphur Elemental Mass Induction Analyser (EMIA series) and a Glow Discharge-Optical Emission Spectroscopy (GD-OES) setup.



## Graphene reactor

A one of a kind modular system for large-scale synthesis of chemically modified graphene, based on chemical graphite exfoliation, is in use. This system is flexible and allows for “on demand” fabrication of materials with tailored properties.

The rig consists 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 is connected to a *Huber Unistat* recirculating chiller.



The manipulation of liquids (e.g. addition of concentrated acids or transfer of slurry between vessels) is carried out using a software controlled peristaltic pump with acid resistant tubing (*Marprene*).

AVA software allows for online control of the temperature in the jacket oil, or the 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... and a purification system based on centrifugation at controlled temperature.

This unique modular approach allows us the flexibility to synthesize materials on demand for different applications.

## Micromechanical Tester

In 2017 CASC purchased a Microtest In-Situ Stage from GATAN, the Mtest300. The tensile/compression/bending stage is primarily design to be used within the confined space of an SEM chamber, although it can be used with optical microscopes, AFM and X-Ray Diffraction machines.

The module allows different materials to be deformed and stretched at loads up to 300N, providing a deeper understanding into what causes the deformation; and the ability to image where the microstructure change is occurring.

## Vibratory Polisher

Before the end of 2018 CASC purchased, with the financial help of the department, a VibroMet 2 Vibratory Polisher from Buehler.

The VibroMet 2 Vibratory Polisher is a machine designed to prepare high quality polished surfaces on a wide variety of materials, including EBSD applications. The 7200 cycles per minute horizontal motion produces a very effective polishing action, providing superior results, exceptional flatness and less deformation. This will be used by most of CASC members.

### **Other equipment**

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

## CASC RESEARCH PORTFOLIO

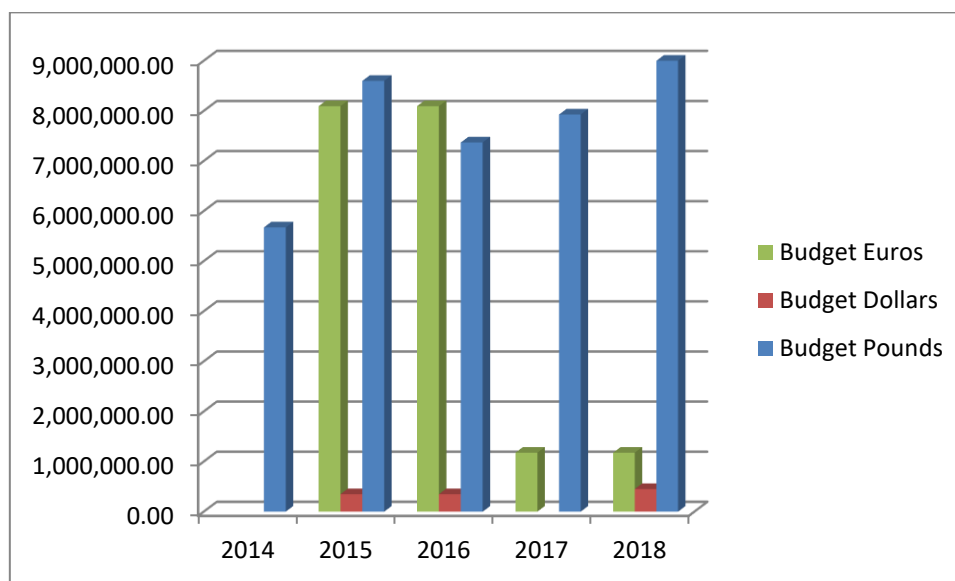


Figure 18. Funded proposals, over the last 5 years

### Current Projects (Jan-Dec: 2018)

- Materials Systems for Extreme Environments.**  
Funder: Engineering and Physical Science Research Council (EPSRC).  
Project start and end date: 2013-2018.  
Budget (£): 3,723,651
- Carbides for Future Fission Environments (CAFFE).**  
Funder: Engineering and Physical Science Research Council (EPSRC).  
Project start and end date: 2015-2019.  
Budget (£): 437,733.
- RESLAG: Turning Waste from Steel Industry into Valuable Low Cost Feedstock for Energy Intensive Industry.**  
Funder: European Commission.  
Project start and end date: 2015-2019.  
Budget (€): 8,092,712, but £300,000 to Imperial.
- Advanced Waste Management Strategies for High Dose Spent Adsorbents.**  
Funder: Engineering and Physical Science Research Council (EPSRC).  
Project start and end date: 2015-2018.  
Budget (£): 191,687.
- High Emissivity Coatings for Furnace Linings.**  
Funder: SCG Thailand.

Project start and end date: 2015-2018.

Budget (£): 497,381.

- **Ceramic Armour from Rice Husk Ash.**

Funder: DSTL.

Project start date: 2016-2018.

Budget (£): 260,000.

- **Transpiration Cooling Systems for Jet Engine Turbines and Hypersonic Flight.**

Funder: Engineering and Physical Science Research Council (EPSRC).

Project start and end date: 2016-2021.

Budget (£): 1,000,000.

- **MAPP: EPSRC Future Manufacturing Hub in Manufacture using Advanced Powder Processes.**

Funder: Engineering and Physical Science Research Council (EPSRC).

Project start and end date: 2017-2023.

Budget (£): 10,000,000, but £750,000 to CASC.

- **AMITIE: Additive Manufacturing Initiative for Transnational Innovation in Europe.**

Funder: European Commission.

Project start and end date: 2017-2021.

Budget (€): 877,500.

- **Ceramic Matrix Composite Technology Development**

Funder: Rolls Royce

Project start and end date: 2018-2021.

Budget (£): 450,000.

- **Wound Care materials**

Funder: Welland Medical

Project start and end date: 2018-2021.

Budget (£): 50,000.

- **Living materials**

Funder: ONRG

Project start and end date: 2018-2021.

Budget (\$): 450,000.

- **Ceramic matrix composites**

Funder: Rolls Royce

Project start and end date: 2017-2020.

Budget (£):175,000.

- **Electrophoretic depositing of graphene on steel**

Funder: ArcerlorMittal

Project start and end date: 2017-2018.

Budget (£):198,000.

- **High Reliability Interconnects: New Methodologies for Lead-free Solders**

Funder: EPSRC

Project start and end date: 2018-2021.

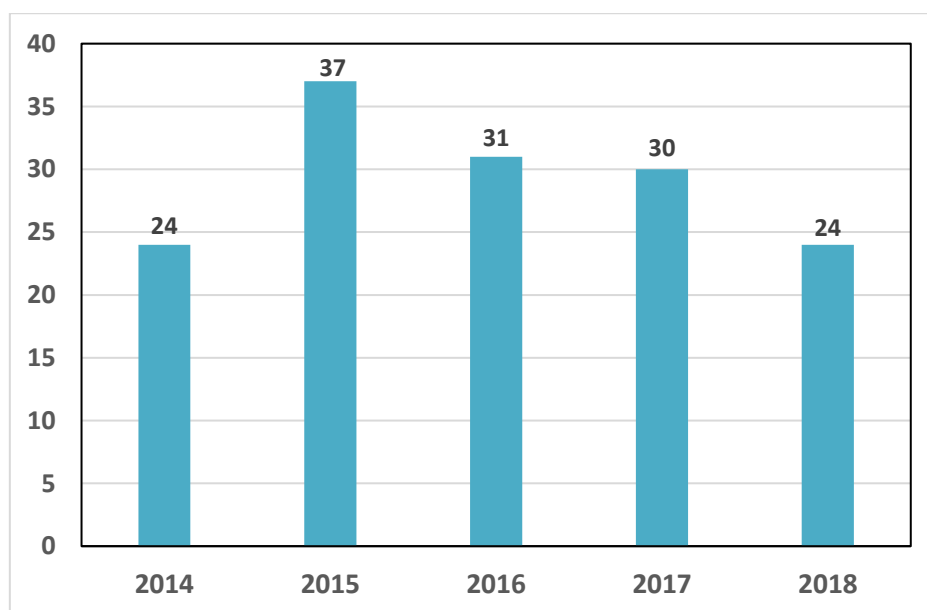
Budget (£): 1,299,714.

- **High temperature deformation and in situ fracture measurements to understand grain size/binder effects in hardmetals**

Funder: SECO Tools

Project start and end date: 2017-2020.

Budget (£): 70,000.



*CASC's number of publications over the last 5 years*

## **Publications: Journal Papers (Jan-Dec: 2018)**

1. *Impact of spark plasma sintering (SPS) on mullite formation in porcelains.* W. Lerdprom, A.Bhowmik, S. Grasso, E. Zapata-Solvas, D. D. Jayaseelan, M. J. Reece, W. E. Lee. *Journal of the American Ceramic Society*, 101 (2), p: 525-535, 2018.
2. *Strong and tough metal/ceramic micro-laminates.* C. Ferraro, S. Meille, J. Rethore, N. Ni, J. Chevalier, E. Saiz. *Acta Materialia*, 144, p:202-2015, 2018.

3. *Temperature-dependent plastic hysteresis in highly confined polycrystalline Nb films.* S. Waheed, R. Hao, Z. Zheng, J. M. Wheeler, J. Michler, D. S. Balint, F. Giuliani. *Modelling and Simulation in Materials Science and Engineering*, 26 (2), 2018.
4. *Deformation behaviour of [001] oriented MgO using combined in-situ nano-indentation and micro-Laue diffraction.* A. Bhowmik, J. Lee, T.B. Britton, W. Liu, T-S. Jun, G. Sernicola, M. Karimpour, D. S. Balint, F. Giuliani. *Acta Materialia*, 145, p: 516-531, 2018.
5. *Calcium phosphate substrates with emulsion-derived roughness: Processing, characterisation and interaction with human mesenchymal stem cells.* G.C. Machado, E. Garcia-Tuñón, R. V. Bell, M. Alini, E. Saiz, M. Peroglio. *Journal of the European Ceramic Society*, 38 (3), p: 949-961, 2018.
6. *SiC porous structures obtained with innovative shaping technologies.* C. Ferraro, E. García-Tuñón, S. Barg, M. Miranda, N. Ni, R. Bell, E.Saiz. *Journal of the European Ceramic Society*, 38 (3), p: 823-8335, 2018.
7. *Editorial on Bioceramics for Bone Repair.* E. Saiz, A. R. Boccaccini, J. Chevalier, M. Peroglio. *Journal of the European Ceramic Society*, 38 (3), p: 821-822, 2018.
8. *Deformation behaviour of [001] oriented MgO using combined in-situ nano-indentation and micro-Laue diffraction.* A.Bhowmik, J. Lee, T. B. Britton, W. Liu, T-S. Jun, G. Sernicola, M. Karimpour, D. S. Balint, F. Giuliani. *Acta Materialia*, 145, p: 516-531, 2018.
9. *Zirconium carbide oxidation: Kinetics and oxygen diffusion through the intermediate layer.* C. Gasparrini, R. J. Chater, D. Horlait, L. Vandeperre, W. E. Lee. *Journal of the American Ceramic Society*. Just accepted.
10. *Data on a new beta titanium alloy system reinforced with superlattice intermetallic precipitates.* A. J. Knowles, T-S. Jun, A. Bhowmik, N. G. Jones, T. B. Britton, F. Giuliani, H. J. Stone, D. Dye. *Data in Brief*, 17, p: 863-869, 2018.
11. *Thermal properties of Cf/HfC and Cf/HfC-SiC composites prepared by precursor infiltration and pyrolysis.* N. Patra, N. Al Nasiri, D. D. Jayaseelan, W. E. Lee. *Journal of the European Ceramic Society*, 38 (5), p: 2297-2303, 2018.
12. *Nacre-like ceramic refractories for high temperature applications.* P.I.B.G.B. Pelissari, F. Bouville, V.C. Pandolfelli, D. Carnelli, F. Giuliani, A. P. Luz, E. Saiz, A. R. Studart. *Journal of the European Ceramic Society*, 38 (4), p: 2186-2193, 2018.
13. *Robocasting of MgO-doped alumina using alginate acid slurries.* D. Glymond, L.J. Vandeperre. *Journal of the American Ceramic Society*, 101 (8), p: 3309-3316, 2018.
14. *In-situ compression and electrochemical studies of graphene foam.* S. Chabi, E. Garcia-Tuñón, H. Chen, Y. Xia, E. Saiz, Y. Zhu. *Veruscript Functional Nanomaterials*, 2: #UMPBGN, 2018.
15. *Production of ceramics from coal furnace bottom ash.* D. Glymond, A. Roberts, M. Russell, C. Cheeseman. *Ceramics International*, 44 (3), p: 3009-3014, 2018.
16. *Durability of hot uniaxially pressed Synroc derivative wasteform for EURO-GANEX wastes.* Y-H. Hsieh, S. A. Humphry-Baker, D. Horlait, D.J. Gregg, E. R. Vance, W. E. Lee. *Journal of Nuclear Materials*, 509, p: 43-53, 2018.
17. *Measuring bone stiffness using spherical indentation.* O. R. Boughton, S. Ma, S. Zhao, M. Arnold, A. Lewis, W. Hansen, J. P. Cobb, F. Giuliani, R. L. Abel. *PLoS one* 13 (7): e0200475, 2018.
18. *Bioinspired Supertough Graphene Fiber through Sequential Interfacial Interactions.* Y. Zhang, J. Peng, M. Li, E. Saiz, S. E. Wolf, Q. Cheng. *ACS Nano*. Just published
19. *“Brick-and-Mortar” Nanostructured Interphase for Glass-Fiber-Reinforced Polymer Composites.* F. De Luca, G. Sernicola, M.S.P. Shagger, A. Bismarck. *ACS Applied Materials & Interfaces*, 10 (8), p: 7352-7361, 2018.
20. *Atomic-scale description of interfaces in rutile/sodium silicate glass–crystal composites.* P. C. M. Fossati, M. J. D. Rushton, W. E. Lee. *Physical Chemistry Chemical Physics*, 20, p: 17624-17636, 2018.

21. *Structure and Properties of High-Hardness Silicide Coatings on Cemented Carbides for High Temperature Applications*. S. Humphry-Baker, J. Marshall. *Coatings*, 8(7), p: 247, 2018.
22. *Layer-by-layer adsorption: Factors affecting the choice of substrates and polymers*. I. S. Elizarova, P. F. Luckham. *Advances in Colloid and Interface Science*, 262, p:1-20, 2018.
23. *Reactive carbothermal reduction of ZrC and ZrOC using Spark Plasma Sintering*. E. Giorgi, S. Grasso, E. Zapata-Solvas, W. E. Lee. *Advances in Applied Ceramics. Structural, Functional and Bioceramics*, 117, 2018.

#### In Press

1. *Ultratough Bioinspired Graphene Fiber via Sequential Toughening of Hydrogen and Ionic Bonding*. X. Wang, J. Peng, Y. Zhang, M. Li, E. Saiz, A. P. Tomsia, Q. Cheng. *ACS Nano*, Just accepted.

## OUTREACH

### Newsletters

CASC's newsletters, together with the annual report, provides news and contact information for visitors to the Centre, together with dissemination at meetings and international visits.

Two newsletters have been circulated in 2018 (June and December), covering new CASC research, visitors to the Centre, PhD thesis defences, the sixth CASC Industry Day, the Sir Richard Brook Prize and the Ninth edition of CASC Summer School, as well as prizes received by different CASC members and media mentions.

Here you can find [June's](#) and [December's Newsletter](#).

Latest news on our activities can also be found through our twitter account (@Casc\_Imperial).

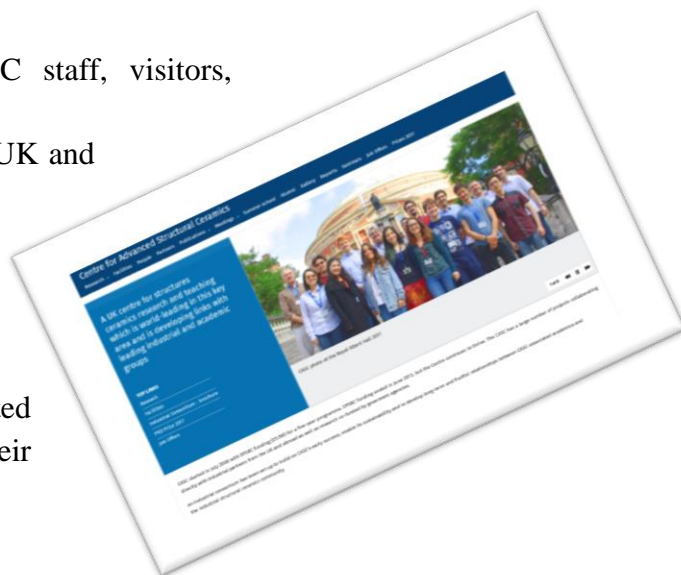
### Website

Our [website](#) contains details of CASC staff, visitors, equipment and activities.

Meeting organised by CASC, as well as future UK and international ceramic-related meetings are also advertised on this website.

Previous annual reports and other publicity material are available for downloading.

The website is continually being updated with new information about CASC staff and their research activities and presentations.



### Sir 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 2017 prize was won by Dr Tahsin Ali Kassam, from Brunel University.

He worked under the supervision of Prof Hari Babu Nadendla on "The Effects of Alumina Purity, TICUSIL® Braze Preform Thickness, and Post – grinding Heat Treatment on the Microstructure, Mechanical and Nanomechanical Properties of Alumina – to – Alumina Brazed Joints". The main goal of his project was to improve the ceramic – to – metal brazing process. The award in general covers a certificate, plaque and £1000 cheque.

The 2018 nominations are closed, and the winner will shortly be announced.

## CASC Industry Day

The seventh CASC Industry Day was held on the 12<sup>th</sup> of January 2018 at Imperial College with different attendees from Industry (Morgan Advanced Materials, John Crane, SAFRAN, SECO, Reaction Engines, Rolls Royce, Lucideon, Johnson Matthey and AMRICC) and University.

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.

In 2019, the meeting will take place the 18<sup>th</sup> of January.



## CASC Summer School on Ceramics

The ninth edition of CASC Ceramic's Summer School took place from the 10<sup>th</sup> to the 12<sup>th</sup> of September 2018.

The first day was focused on **mechanical properties of Ceramics** and their use in **Thermal Barrier Coatings and Solid Oxide Fuel Cells**, with the talks of **Dr Finn Giuliani** (*Imperial College*), **Professor Ping Xiao** (*Manchester University*) and **Professor Alan Atkinson** (*Imperial College*).

On Tuesday, we also had the visit from some of the members of the **Additive Manufacturing Initiative for Transnational Innovation in Europe (AMITIE)** project as the day was focused on **Additive Manufacturing**. For this, we had a **masterclass on the basics of additive manufacturing** by **Stephane Richaud** (*Saint Gobain*) and **Dr Andrea Zocca** (*BAM University and Young Ceramists Additive Manufacturing Network*) together with **two lab sessions** focused on 3D Printing and on the demonstration of some of the mechanical properties discussed on the first day.

We finished the ninth edition of the Summer School with the invited talk of **Dr Rachid M'Saoubi** (*SECO Tools*) that gave us some **industrial examples of the use of ceramics**.

We hope the attendees enjoyed their stay here at Imperial College and hopefully we will see them at the **10<sup>th</sup> edition!**.



The course was attended by a mix of people from industry (Morgan Advanced Materials, Saint Gobain, Rolls Royce,) and academy (Imperial College, ETH, Queen Mary University).

The exact date and programme of the next edition will be published early in 2019, after input from our industrial consortium.

### **Meetings attended during 2018**

- 42<sup>nd</sup> International Conference and Expo on Advanced Ceramics and Composites, Daytona Beach, USA.
- MAPP First International Meeting, Sheffield, UK.
- RESLAG Project meeting, Grenoble and London.
- 9th International Workshop on Interfaces. New Frontiers in Biomaterials, Santiago de Compostela, Spain.
- Quarterly MAPP meeting, Imperial College and Oxford, UK.
- Young Ceramists Additive Manufacturing Forum, Padua, Italy.
- 14<sup>th</sup> International Ceramics Congress, Perugia, Italy.
- 7<sup>th</sup> International Congress on Ceramics, Foz do Iguazu, Brasil

- UK-Israel Microscopy Summer School, Israel.
- 10<sup>th</sup> European Solid Mechanics Conference, Bologna, Italy.
- AMITIE workshop, London, UK.
- International Colloquium Refractories, Aachen, Germany.
- Small-Scale Properties of Materials and Length-scale Phenomena symposium at the MS&T 18, Ohio, USA.
- 10<sup>th</sup> International Conference on High Performance Ceramics, Nanchang, China.
- 2018 MRS Fall Meeting & Exhibit, Boston, USA.