

Centre for Advanced Structural  
Ceramics  
2020 Annual Report

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## Introduction

CASC has been continuously running since it started in July 2008 with EPSRC funding (£5.5M) for a five-year programme. After the end of the EPSRC funding in June 2013, but we have kept growing each year, establishing new industrial and academic collaborations both abroad and in the UK and participating in numerous national and international initiatives. This year a large number of PhD students have started working with us on a wide range of research projects, from additive manufacturing to mechanical testing.

This has been a difficult year for all. Our laboratories were closed for several months and some of our activities stopped including our Ceramics Summer School. Our laboratories opened again in the second half of the year and we have re-started our dissemination and training activities. We have put measures in place to provide a safe working environment. We have also secured new projects that will bring new equipment to the Centre, including a new Spark Plasma Sintering system in an ERC grant won by Dr. Florian Bouville.

The centre collaborates with Industry in several projects and maintains an Industrial Consortium to 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.



## 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 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 17th of May of 2011, where it was well received by the industry representatives and was developed by our Steering Group on July 4th 2011.

The Industrial Consortium started functioning in 2014 after the Steering Group meeting, held the 17th 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:

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

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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>.

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

## People

### Staff

#### Professor Eduardo Saiz



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  $Ti_3SiC_2$ , which combine ceramic and metallic properties. However, of particular interest is their ability to dissipate energy through reverse plasticity. This continues to be a topic of research.

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  $Mg_2SiO_4$ .

#### **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. Iuliia Elizarova</b>	Research Associate
<b>Dr. Oriol Gavalda Diaz</b>	Research Associate
<b>Dr. Siyang Wang</b>	Research Associate
<b>Dr. Madeleine Watson</b>	Research Associate

## PhD Students

<b>Kareem Abdelhadi</b>	1 <sup>st</sup> year
<b>Mercedes Baxter</b>	1 <sup>st</sup> year
<b>Qiasong Cai</b>	3 <sup>rd</sup> year
<b>Aaron Chote</b>	1 <sup>st</sup> year
<b>Hugh Collett</b>	1 <sup>st</sup> year
<b>Ben Currie</b>	2 <sup>nd</sup> year
<b>James Davidson</b>	1 <sup>st</sup> year
<b>Justine Delage</b>	3 <sup>rd</sup> year
<b>Max Emmanuel</b>	2 <sup>nd</sup> year
<b>Yinglung Hong</b>	1 <sup>st</sup> year
<b>Tianhui Jiang</b>	1 <sup>st</sup> year
<b>Jindaporn Juthapakdeeprasert</b>	3 <sup>rd</sup> year
<b>Ming Li</b>	1 <sup>st</sup> year
<b>Jack Lyons</b>	2 <sup>nd</sup> year
<b>Ollie Osborn</b>	1 <sup>st</sup> year
<b>Harry Payne</b>	1 <sup>st</sup> year
<b>Jia Hui Teo</b>	3 <sup>rd</sup> year
<b>Victoria Vilchez</b>	1 <sup>st</sup> year
<b>William Wei</b>	1 <sup>st</sup> year
<b>Kathryn Yates</b>	3 <sup>rd</sup> year
<b>Shitong Zhou</b>	2 <sup>nd</sup> year
<b>Muhammed Maktari</b>	2 <sup>nd</sup> year

## Research

### Research Fellow Projects

**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.

**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**

### ***Additive Manufacturing of Ceramics and Composites***

#### ***Dr. Iuliia S Elizarova***

My research is concerned with additive manufacturing, mainly robocasting, of ceramic materials and composites, and exploring the capabilities of the technique in terms of versatility and applications. Robocasting produces parts by continuous extrusion of powder-based pastes in a layer-by-layer manner. Composition of such pastes is determinant to the quality of the printed parts, as well as it defines the resolution and complexity of the prints. The shear forces present in the nozzle during printing can be used to align the particles composing the inks if such particles are anisotropic in shape (fibres, platelets), which, in turn, allows for tailoring of mechanical properties of the created structures (fabrication of composites). On the other hand, fabrication of geometrically complex parts with presence of overhanging or free-standing features is a limitation of the technique (due to its layer-by-layer nature that requires supporting layers for the newly printed ones), which can also be aided with formulation of the pastes (ones that, for example, allow for post-printing shaping of

the parts). Expansion of applicability of robocasting, whether it's through development of the technique itself or stock materials, is, therefore, the aim of my research activities.

### **Environmental degradation of SiC/BN/SiC Ceramic Matrix Composites for aerospace applications**

**Dr. Oriol Gavaldà Diaz**

The need to increase the cycle efficiency and reduce NOx emissions from aero-engines has promoted the development of Silicon Carbide (SiC) based Ceramic Matrix Composites (CMCs) which have entered in service in aircraft turbine engines as replacements for some Ni-based superalloys. The main tendency of material choice is converging to CMCs constituted by SiC fibres coated with a thin (0.1-1 μm) BN interphase within a SiC matrix (SiC/BN/SiC), resulting in an optimised tough ceramic composite. However, unlike the generic tendencies found for metallic materials, environmental effects seem to not follow a clear tendency as hottest temperatures do not necessarily result in more severe degradation. This is due to the complex degradation thermodynamics occurring at the interface of the SiC-BN system such as volatilisation of B species, borosilicate glass formation or formation of self-healing oxide products.

An important aim of this projects consists in understanding how the interfacial and fibre properties in SiC/BN/SiC are affected by different aero-engine inspired degradation cycles by exploring the interfacial properties via push-out tests and the fibre properties via three point bending of single fibres (see Figure 1). Further TEM-based characterization techniques are done in order to link the change in properties to the changes in chemistry and microstructure.

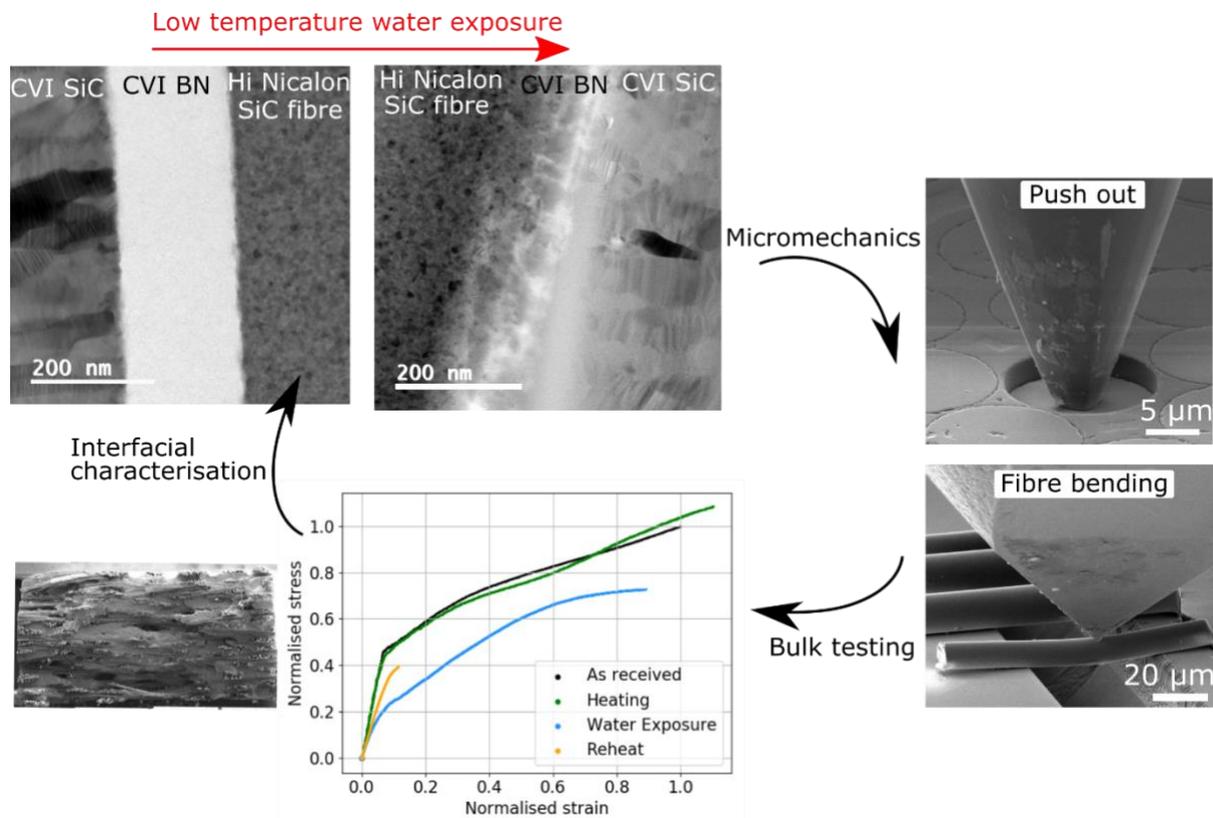




Figure 1 Schematic showing the methodology used to characterise the degradation of CMCs: (i) STEM analysis to understand the change in morphology and chemistry at the interfacial region, (ii) micromechanics of the interface and fibres and (iii) fractography after tensile bulk testing.

**Dr Siyang Wang**

***Optimising the Performance of Magnetocaloric Materials.***

Cooling applications uses 15% of the world energy consumption. Solid state cooling materials, such as magnetocaloric materials, offer the possibility to be far more efficient than their conventional gas compressor counterparts and therefore drastically reduce CO<sub>2</sub> emission. The major challenge is to successfully incorporate magnetocaloric materials in to functioning cooling systems with good mechanical stability. The aim of this project is to work with our industrial partner Camfridge to optimise the performance of magnetocaloric materials so they can be incorporated into a working prototype. In this project, we will perform research into the in operando behaviour of magnetocaloric alloys (La-Fe-Si based materials), to understand mechanisms of failure, and to design materials processing routes to optimise performance under active cooling conditions. The project is in collaboration with Camfridge UK and is funded by Innovate UK.

**Dr Madeleine Watson**

***Transpiration Cooling to Enable Sharp Leading-Edge Technology***

***Sponsor: EPSRC***

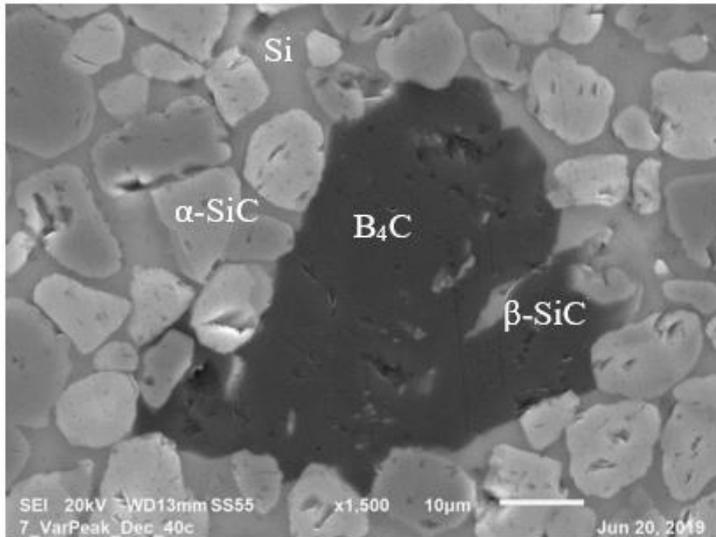
Transpiration cooling has long been proposed as a thermal protection system for sharp leading edges on hypersonic vehicles. During operation, these parts are exposed to temperatures in excess of 2000 °C, which makes high melting Ultra-High Temperature Ceramics (UHTCs) lead candidates fabricating such parts. However, UHTCs are generally Borides and Carbides, which tend to convert to much lower melting oxides under the plasma heading conditions created in hypersonic flight. Working with a team of hypersonic engineers at the University of Oxford, this project aims define parameters for generating protective layers of laminar flowing inert gasses across the surface of leading-edge parts.

Currently, we aim to probe the relationship between gas flux, laminar flow layers and surface concentrations of oxygen species. From a materials standpoint, engineering laminar flow layers requires materials with very consistent distributions of open porosity, which in the past we have successfully achieved by partially sintering 3-5µm particles of the UHTC ZrB<sub>2</sub> to approximately 65% density. However, the easiest way to quantify local oxygen concentrations is with surface bound oxygen-responsive fluorophores, also known as oxygen partial- pressure sensitive paint (PSP), and PSP does not stick well to ZrB<sub>2</sub>. Luckily, PSP sticks well to Alumina. Toward this aim, we have produced partially-sintered porous alumina from size matched alumina feedstocks, allowing us to visualise local surface oxygen concentration as a function material density and transpiring gas pressure.

## PhD Projects

### **Kareem Abdelhadi**

Silicon Carbide ceramics exhibit high temperature oxidation behavior. This makes them promising candidates for the gas turbine hot section. However, at high temperatures, and water vapour, SiO<sub>2</sub> layer becomes unstable and volatilizes. This limits the operating temperature of gas turbines. To increase gas turbine efficiency, and



limit carbon emissions, higher combustion temperatures are needed. Thus, Environmental Barrier Coatings (EBC) are required to protect SiC substrate from degradation, and recession. Rare-Earth (RE) EBCs have been identified as ideal candidates, but they are susceptible to hot corrosion in the combustion environment, and hence their durability is limited. My research aims to understand the following (a) oxidation behaviour of

Reaction Bonded SiC-B<sub>4</sub>C (RBSBC), and (b) the corrosion behaviour of RE-oxide EBCs (Yb, Er, and Lu silicates) processed on RBSBC using dip coating. The thermochemical and thermomechanical behaviour of RE-EBCs are investigated under hot corrosion environment simulating the corrosive conditions in gas turbine hot section. Characterization using Scanning Electron Microscope equipped with Energy Dispersive Spectroscopy, X-Ray Diffraction, Transmission Electron Microscopy, and Raman spectroscopy are used to develop modelling methodology to predict the life and durability of EBCs and understand its corrosion, and fracture, behavior.

### **Mercedes Baxter**

The fuel ponds at Sellafield contain a brucite-rich sludge, with a pH of 9 – 10. The focus of the PhD is on storing the sludge and making it safe, by converting the waste to a magnesium silicate hydrate (M-S-H) based binder.

Magnesium silicate hydrate cements are a relatively new type of binder that can be made using brucite (or MgO) and soluble silica to form an amorphous M-S-H gel. This has a pH of 9.5 – 10.5, which is compatible with other products also contained in the sludge (e.g. Al, Mg). M-S-H therefore offers a potential for waste volume reduction and immobilisation, as the sludge would effectively be part of the stabilising cement. The long-term durability and behaviour of M-S-H gel is largely unknown.

The overall aim of the research is to determine how the sludge should be pre-treated to produce a suitable M-S-H based wasteform and to characterise its long-term durability. This will be achieved by creating an artificial sludge like material by slow formation and sedimentation of Mg(OH)<sub>2</sub>. The simulated sludge will then be processed

to produce M-S-H based wasteforms. These M-S-H based wasteforms will then be studied, focusing on the composition, microstructure, mechanical and durability properties at different ages and under varying conditioning regimes.

**Qiaosong Cai**

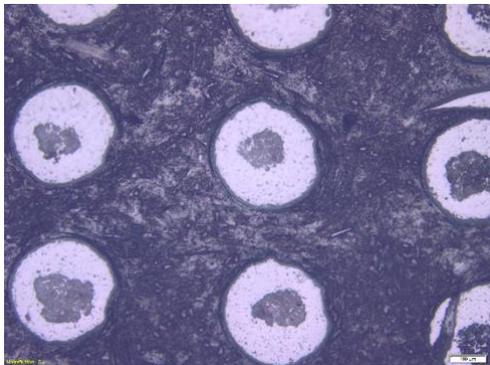
**Robocasting of complex structural ceramics**

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

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 here are emulsion-based inks in which the macroscopic shape and microscopic porosity of the objects can be controlled easily.



**Aaron Chote**

Zircaloy-4 (Z4) is the principal alloy used in fuel cladding in the nuclear industry. Zirconium has a high affinity for oxygen at high temperatures and consequently instantaneous oxidation at the surface of zirconium creates an oxide layer. The oxide growth process occurs in three stages and changes the properties of the layer. Initially the oxide layer acts as a protective barrier slowing down corrosion (the pre-transition state). The layer further evolves to a stage where hydrogen and oxygen transport occur through defects created as a result of increased mechanical stresses (post-transition).

The  $ZrO_2$  oxide layer consists of two polymorphs (monoclinic and tetragonal) that exist in differing amounts dependent on the transition regime; tetragonal dominates in the pre-transition and monoclinic in post-transition.

To understand the influence of interfaces (grain boundaries and phase boundaries) distribution in the oxide layer, precession electron diffraction (PED) is required, as the grains are often on the scale of a couple of nanometres. PED will yield grain orientation data that we will analyse to obtain the grain boundary plane distribution (GBPD) in the growing oxide layer. Our evaluation will provide quantitative information on which interfaces are the most frequent ones, as opposed to qualitative information.

### **Hugh Collett**

#### **Saving the Mary Rose: Determining the structural and material properties of a Tudor warship for future preservation.**

My project is focused on investigating the behaviour of the wood of the Mary Rose ship, a 500 year old Tudor warship. The material composing the ship is a complex mixture of archaeological wood and polyethylene glycol, added as a consolidant to strengthen and prevent the shrinkage associated with drying wet wood. As such, it is not a well understood material and the properties must be determined to prevent further degradation. A combination of computational and experimental methods will be used to determine engineering properties such as Young's modulus and yield strength, which will be included in a finite element model. The model will form the basis of structural analysis, necessary to identify critical zones in the hull and pinpoint areas requiring increased structural support.

### **Ben Currie**

Whilst tungsten monocarbide (WC) has been used extensively in the tooling industry within hard metals, use of monolithic WC has remained minimal. This has led research to be focused on the hard metals themselves, whilst the properties of monolithic WC have remained relatively unstudied. Recently there has been renewed interest in the research of monolithic WC due to its promise as a neutron shielding material in compact spherical tokamaks. This application will expose the shielding material to an environment of extremely high temperatures and neutron fluxes. Whilst monolithic WC is of interest due to its high chemical and thermal stability, low activation properties, high attenuation of both gamma and neutrons as well as high hardness and sputtering resistance, its high temperature mechanical properties are still not fully understood and its mechanical properties after irradiation are yet to be studied at all.

This project therefore aims to use techniques such as compressive creep testing, compressive yield testing, mutual indentation and nanoindentation in conjunction with ion implantation to study the effects of the fusion environment on monolithic WC, expanding on the literature and assessing its suitability as a neutron shielding material.

### **James Davidson**

Development of smaller fusion reactors presents several challenges to the design, primarily the proximity of the fusion plasma to the magnets that confine it. Unlike in



standard reactor designs, there is only a small space in which the shielding materials need to be placed, therefore highly effective shielding materials are required. Currently, most conventional shielding materials are unable to provide adequate neutron shielding efficiency to the toroidal magnets, leading to increased degradation and heat deposition, reducing lifetime and performance.

Simulations work has shown that ceramic compounds in the tungsten-boron system are able to provide high levels of neutron shielding. This is due to the high scattering cross section of the tungsten and the high neutron capture cross section of boron.

While the shielding properties of the W-B system are ideal, the mechanical properties make manufacturing an issue due to their brittle nature leading to high hardness and low toughness. In order to improve these properties, composites manufactured with mixtures of pure tungsten and tungsten borides will be investigated in this project. One aspect of this work will be modifying the W-WB composite using several thermomechanical processing techniques to improve the toughness. The focus of this work will be to see how these thermomechanical processing techniques affect the microstructure and to relate these changes to the mechanical properties of the W-WB composite.

**Max Emmanuel**

**Study of grain boundary character and strength in WC-Co**

**Supervisor: Dr. Finn Giuliani and Dr. Ben Britton**

**Sponsor: Seco**

Description of the project: The aim of the project is to examine the role of microstructure and binder chemistry on interface properties in crack growth. Different types of WC/WC grain boundaries (in terms of CSL) making up WC-Co have been identified. Work is being done to fabricate double cantilever beams (DCB) around these boundaries with the intent of understanding interface properties. DCB tests have been performed on WC single crystals and large WC grains (~20-100  $\mu\text{m}$ ) as a basis for interface study: this is to be used as an opportunity to determine the surface energies of WC crystallographic planes and compare with density functional theory calculations from the literature. Future work will be concerned with studying WC/WC boundaries with cobalt infiltration or chromium layers. WC/WC cobalt infiltration occurs during creep due to grain boundary sliding. Chromium is added to inhibit grain growth.

**Yinglun Hong**

**Self-healing Epoxy Composite**

The main target of this project is to design epoxy composites which exhibit sensing ability, excellent mechanical strength, self-healing and shape memory features. The thermally responsive self-healing and shape memory epoxy vitrimer was presented by IK4-CIDETEC research centre. This epoxy exhibits fast stress relaxation above the glass transition temperature due to the aromatic disulphide exchange reactions. However, this material is not strong enough for load-bearing applications and cannot sense the environment (electrical and mechanical stimuli). In this project the approach will be divided into the following aspects:

1. Testing the self-healing and self-shaping abilities of the epoxy resin. Improve the formulation of the epoxy to achieve better performance.
2. Design polymer-ceramic composites with brick-and-mortar structure to enhance the mechanical strength of the epoxy resin. The micro alumina platelets will be employed to be the ceramic "bricks". The mortar will be a mixture of graphene and epoxy. Graphene is added to form a conductive interconnected network. Therefore, the composite can respond to electrical stimuli. In this way the material can also monitor the formation of damage.
3. The possibility of processing the composites by robocasting technique will be studied.

### ***Tianhui Jiang***

The aim of my project is to develop a ceramic composite anode material with high capacity and fast lithium ion conduction path for energy storage devices. The project includes the design of thermally responsive ceramic slurries for robotic assisted 3D printing and pH responsive slurries for gel casting. So that fabrication of composite with designed ink can be achieved. Structures and properties of designed material will be studied by applying a range of characterization techniques (rheology test, electron microscopy...).

On the other hand, functionalization of lithium ion conducting particles will be studied to optimize the microstructure of the composite. Super paramagnetic iron oxide nanoparticles will be coated onto the particle surface to make them responsive to magnetic field. Hence, magnetic templating can be used to modify the microstructure of anode material to achieve the fast lithium ion conduction in anode with high capacity by aligning the conducting particles in material matrix.

The final goal is to develop designs and manufacturing processes that will lead to a new generation of more efficient energy storage devices.

### ***Jindaporn Juthapakdeeprasert***

The world faces probable catastrophe due a significant increase in energy consumption and CO<sub>2</sub> gas emission in coming decades. The cement industry is in the top 5 contributors to this crisis. Unfortunately, in the cement rotary kiln, 40% of the input energy is lost as heat. Nearly half of that is lost through the shell of the kiln. Reducing this lost energy is a challenging topic for a PhD research project. My PhD focuses on developing a high emissivity coating for the inside surface of the cement rotary kiln. The function of the coating is to absorb heat then re-emit it back inside instead of being lost to the environment through the refractory brick and kiln shell (see figure below). The closer the emissivity value is to a value of 1 the more energy is saved. The coating being studied is composed of cerium oxide, a most abundant rare earth element with high melting point (2477°C) and high emissivity (0.9 at 1000-2000°C). The coating contains a phosphate-based binder to give adhesive force between the cerium oxide and the substrate. The substrate and coating emissivities were measured with an in-house emissivity apparatus at Center National De La Recherche Scientifique (CNRS), Orléans, France. They reveal that a substrate coated with high emissivity designed coating gave an emissivity value of 0.8 double the emissivity of an



uncoated substrate which was 0.4. In conclusion, this research reveals a promising high emissivity coating that can improve the cement rotary kiln efficiency and reduce damage to the world's atmosphere.

### **Ming Li**

Containing the development of new processing techniques for the fabrication of ceramic-based intelligent manipulation materials, self-healing & controllable composites, and super-hydrophobic / oleophobic surface, in particular hierarchical composites with bioinspired architectures.

### **Jack Lyons**

My research has focused on understanding the fundamental formation mechanisms and properties of the MAX phases, a group of nanolaminate ternary carbides/nitrides. The project has been split into three key areas formation mechanisms; micromechanics and tuneable plasticity of a ceramic. All three sections inter-connect so initially the project focused on trying to create phase pure samples of  $Zr_3AlC_2$ , for analysis, however this proved difficult so investigations began to understand the formation mechanism of this material through the use of In-situ and ex-situ XRD experiments at different temperatures to track the reaction. This was done so that firstly a better understanding of the formation mechanism should allow for creation of phase pure/as close as we can get to phase pure samples and secondly when trying to tune the plasticity through doping of the material we would be able to understand at which point the dopants could be included. The MAX phases for being partially ceramic are a relatively soft material which is still not fully understood, therefore a combination of micromechanics experiments such as micro-fracture and micro-pillars and spectroscopic techniques are being used to probe these properties. Most recently the correlative use of EBSD and Raman has been investigated to allow for potential strain and orientation analysis of the MAX phases using Raman. This is done as Raman has the added benefit of being able to probe the different layers of the MAX phase (ceramic and metallic) at once as it is a vibrational based techniques and the MAX phases contain two different sets of bonds. There are not many other techniques that allow you to probe at the atomic level without much greater time/effort required. So as we greater understand the formation mechanisms of these materials, we can start to introduce dopants to alter their properties and then characterise these through a combination of micromechanics and spectroscopic techniques."

### **Ollie Osborn**

One of the major limitations of traditional ceramic processing methods is their inability to manufacture parts with complex geometries. Attempts to overcome this problem has shifted the attention of industry to additive manufacturing (AM). The project focuses on the AM of ceramic carbides, predominantly silicon carbide and boron carbide, and their composites. The techniques of interest are selective laser sintering (SLS) and a novel method based on photolithography. Both these methods can produce complex shapes, but the material properties of the final ceramic parts currently limit them to prototyping. The aim of the project is to overcome this limitation

and produce complex parts with material properties that allow for industrial applications. The project is in collaboration with The Manufacturing Technology Centre (MTC) and MAPP.

**Harry Payne**

Boron Carbide (B4C) is well known for its' hardness and has been a material of great interest for the defeat of ballistic threats. However, above a critical pressure, the B4C undergoes phase collapse, hence losing its ballistic properties. Silicon Carbide, on the other hand, does not undergo phase collapse during impact but is denser and expensive to produce. Alumina, the base of most ballistic armours, is the densest of this trio and relatively inexpensive. Alumina's properties, whilst fine for current threats, are not optimised for ballistic armour and improvements can be made to performance and cost. As the military equipment grows in weight, the need for a light and effective armour is obvious. The aim of this project is to see if the properties of B4C and SiC can be combined by producing a nanocomposite that is light, hard and has sufficient toughness to enhance ballistic and multi-hit performance. This is a ground-up project, sponsored by the Defence Science Technology Laboratory [DSTL], considering all stages of production and property selection.

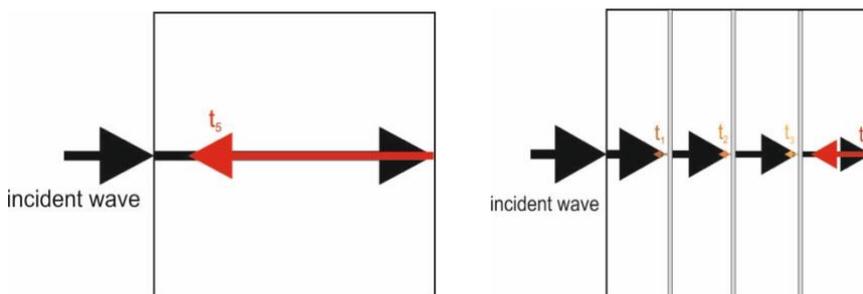
**Jia Hui Teo**

**Impedance engineering for better armour ceramics**

**Supervisors: Prof Luc J. Vandeperre and Prof Eduardo Saiz**

Projectile erosion accounts for a significant loss of energy during projectile-target interaction, and the duration is proportionate to the amount of time the ceramic can maintain its integrity[1]. Literature review suggests that the dwell time of the projectile on the surface is directly correlated to the thickness of the tile instead of areal density or density.

This thickness dependence is that failure of the ceramic tiles during projectile impact is largely due to the tensile reflection arising from an impedance mismatched interface. A projectile impacting a ceramic tile surface constitutes a compressive wave. In a monolithic ceramic, the only tensile reflection is from the back end of the ceramic tile. Due to the large impedance mismatch between the ceramic tile and air, this reflection arising from the ceramic/air interface is very large and is problematic as ceramics are weak in tension. By introducing interfaces with slight impedance mismatches, the magnitude of the large tensile wave arising will be replaced with many smaller tensile returns which will improve the survivability of the tile albeit only by a couple of microseconds.





**Figure 2: Schematic of reflected wave from a monolithic ceramic (left) and from impedance engineered composite with interfaces with small impedance mismatches.**

### **Victoria Vilchez**

#### **Development of hierarchical reinforcements in bioinspired ceramics**

One of the main limitations in the use of ceramics comes from their intrinsic brittleness which makes them very sensitive to defects and prone to catastrophic failure. Natural materials (nacre, bone, dentin) have found ways around this problem by adapting their microstructure at multiple length scales. Such structural hierarchy provides failure mechanisms that increase damage tolerance by several orders of magnitude in comparison to the bulk ceramic they are based upon.

Starting from seashell's structure blueprint, nacre-like ceramics and composites have recently been developed and can now get properties on par with some of the state-of-the-art composites used in aeronautics. The objective of this project is to characterise the fracture mechanisms in the newly developed materials from the micrometre to the millimetre scale. Based on these results, the next steps will be to rationally design the next generation of bioinspired ceramics by further adapting the microstructure to induce new toughening mechanisms.

### **William Wei**

William's project aims to study the adhesion strength of environmental barrier coatings (EBCs) applied on silicon carbide (SiC) ceramic matrix composites (CMCs) to develop the next generation of gas turbines that are faster, cheaper, lighter, more efficient and less pollutant. And he is currently working on two areas;

- Developing mechanical testing methodologies that will enable an accurate and reliable characterization of the adhesion strength at the coating/CMC interface using the single layer approach.
- Combining systematic mechanical testing and structural characterization in order to develop a deep understanding of the parameters that control adhesion, delamination, spallation and the models needed to support the design of new materials.

### **Kathryn Yates**

**Supervisors : Dr Finn Giuliani, Dr Katharina Marquardt**

**Industrial Sponsor: AWE**

I am investigating self-irradiation of plutonium using surrogate materials which have been subject to radiation effects. The UK has stockpiles of plutonium from our energy and defence programmes, and further knowledge of how the material ages is desirable for safe-handling, use, and long-term storage. Radioactive decay of plutonium results in lattice damage, compositional changes, and helium accumulation due to  $\alpha$ -decay. These phenomena are thought to affect the materials mechanical behaviour, corrosion behaviour and phase stability. In this study surrogate materials are irradiated with helium ions and subject to lattice damage using ion accelerators at the Dalton Cumbria Facility, and samples are analysed using TEM techniques including EELS and *in-situ* heating, and micromechanical testing.

**Shitong Zhou**

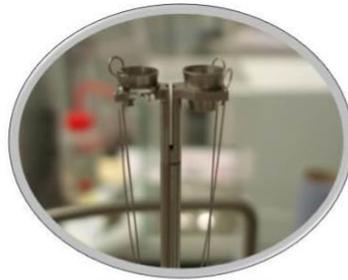
***Embedded 3D printing Multimaterial composites on ceramics***

***Supervisors: Prof Eduardo Saiz***

***Sponsor: CASC Industrial Consortium***

The project is to investigate embedded 3D printing multimaterial composites on ceramics, especially printing fibers inside ceramics and complex structures. The chosen healable matrix for printing is a Pluronic-based slurry. The viscosity of this slurry is sensitive to the temperature, which has good mobility at low temperature, so it flows around the fibers and fills the voids between fiber and matrix. As an initial proof-of-concept, steel fibers are printed in Alumina slurry and the effect of rheological behavior in this system has been investigated. Drying is the biggest problem in this kind of hydrogel-based system and will be improved by a deep investigation of tomography during the drying period. And microstructure of the samples after sintering will be analyzed and tailored to improve the interface between fibers and matrix and the mechanical performance. This kind of embedded 3D printing will be further applied on other materials to broadening the application.

## Capabilities and Facilities



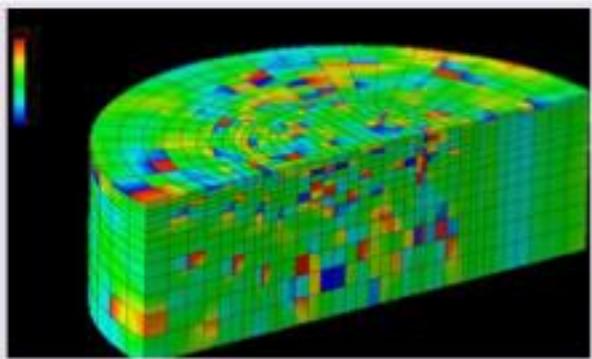
Although the purchasing and installation of large pieces of equipment by the Centre using the funding from the original CASC project is now completed, we continue to improve our experimental capability in this area using funds from other sources.

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).

### 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.

### Freeze dryer



Freeze-drying is a drying process, where the solvent (normally water) is eliminated from the sample via direct sublimation from solid to gas phase. This is a useful way 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.

### 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.

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 ZrB<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> and the analysis of cracking due to shrinkage in geopolymers and sintering of silicon carbide-boron carbide composites.

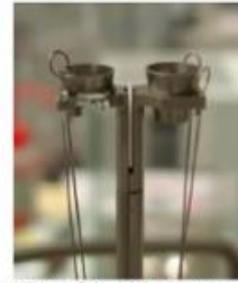
The **combined TGA-DTA** has been used to quantify mass loss during drying of geopolymers, to analyse the decomposition of magnesium phosphate and magnesium silicate cements for nuclear waste treatment, to study silicon carbide or mullite sintering, to perform analysis of UHTC oxidation, to determine carbon yield from various ceramic additives and for characterisation of raw materials in general. Usage for third parties included work with Professor Jon Binnner (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 ranged of the merasurement up to 1650 oC 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 2000oC, the second frame has induction heating up to 1200oC.



Crucibles of the high temperature combined TGA DTA

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 led 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 gases.

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<sup>-2</sup> to 10<sup>-4</sup> μm 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 1750oC 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 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.

CASC also has two Digital Light Processing systems for additive manufacturing of ceramics and polymers the Liquid Crystal Ceramic Precision 1.5 and the Liquid Crystal Precision 1.5 from Photocentric.

### ***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.

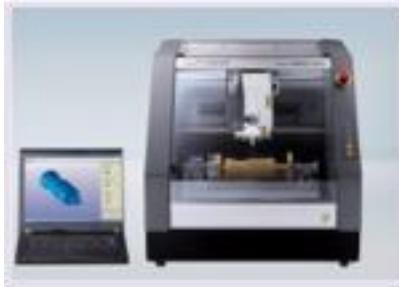


It is a three-part system: Extruder with Take Up, and Chopper Accessories. Maximum temperature 400°C and Variable speed control, 5 to 260rpm.

### **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**

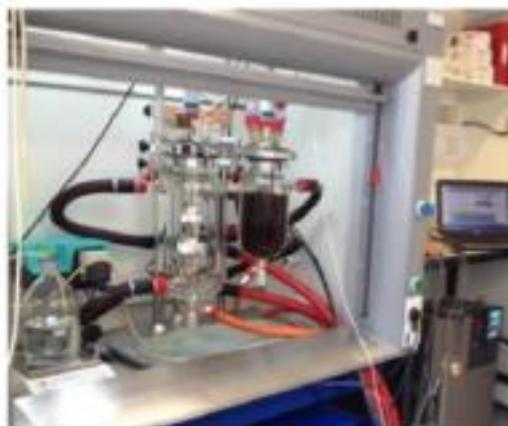
An Oxygen-Nitrogen-Hydrogen Elemental Mass Gas Analyser (Horiba, EMGA – 830 series) was installed at CASC in 2015. This includes also the Carbon-Sulphur Elemental Mass Induction Analyser (EMIA series) and a Glow Discharge-Optical Emission Spectroscopy (GD-OES) setup.

**Graphene reactor**

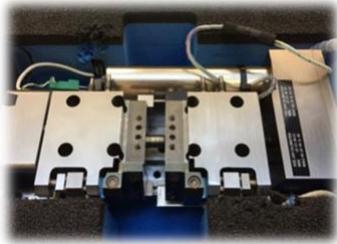
A one of a kind modular system for large- scale synthesis of chemically modified graphene, based on chemical graphite exfoliation, is in use. This system is flexible and allows for “on demand” fabrication of materials with tailored properties.

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.

Recently we have upgraded our capabilities with a Gatan Microtest 2kN (MTest 2000E) loading frame (tensile/compression/bending) with heating up to 500°C specifically for in situ SEM/EBSD experiments.

### **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.

### **Automated cutting machine**



The CASC purchased, with the help of the department, an Accutom 100 from Struers. This saw is capable of cutting hard using diamond coated bladed but also to do programmable cuts. This allows the preparation of specimen with precisely controlled and repeatable sizes, for preparing specimen for mechanical testing for instance. In addition, this machine is capable of accurately grind and flatten the surface of samples using a diamond coated cup wheel, producing flat and parallel surfaces.

### **Other equipment**

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



## CASC Research Portfolio

### Funded Proposals Starting in 2020

Renewal of the MAPP EPSRC processing hub (£1.4M)

Eduardo Saiz signed a collaboration with Jiangsu Delin Environment Protection Technology to develop ceramic microfluidic chips to test water quality (£25K)

Florian Bouville received an ERC Starting grant (£1.2M) to pursue research into novel toughening mechanisms for brittle materials over the next 5 years.

Finn Giuliani and Katharina Marquardt from CASC along with other researcher in the materials' department received an EPSRC large equipment grants (£10,2M) to fund a new high resolution and environmental electron microscopy suits.

Luc Vandeperre received a grant (£385K) from DARPA to study transpiration cooling

### Publications

Diaz OG, Marquardt K, Harris S, et al., 2020, Degradation mechanisms of SiC/BN/SiC after low temperature humidity exposure, *Journal of the European Ceramic Society*, Vol: 40, Pages: 3863-3874, ISSN: 0955-2219

McGilvery C, Jiang J, Rounthwaite N, et al., 2020, Characterisation of carbonaceous deposits on diesel injector nozzles, *Fuel: the science and technology of fuel and energy*, Vol: 274, Pages: 1-9, ISSN: 0016-2361

Jones LD, Vandeperre LJ, Haynes TA, et al., 2020, Theory and application of Weibull distributions to 1D peridynamics for brittle solids, *Computer Methods in Applied Mechanics and Engineering*, Vol: 363, Pages: 1-11, ISSN: 0045-7825

Zhou T, Wu C, Wang Y, et al., 2020, Super-tough MXene-functionalized graphene sheets, *Nature Communications*, Vol: 11, ISSN: 2041-1723

Haug M, Bouville F, Ruiz-Agudo C, et al., 2020, Cold densification and sintering of nanovaterite by pressing with water, *JOURNAL OF THE EUROPEAN CERAMIC SOCIETY*, Vol: 40, Pages: 893-900, ISSN: 0955-2219

Bhowmik A, Lee J, Adande S, et al., 2020, Investigating spatio-temporal deformation in single crystal Ni-based superalloys using in-situ diffraction experiments and modelling, *MATERIALIA*, Vol: 9, ISSN: 2589-1529

Elizarova I, Vandeperre L, Saiz Gutierrez E, 2020, Conformable green bodies: plastic forming of robocasted advanced ceramics, *Journal of the European Ceramic Society*, Vol: 40, Pages: 552-557, ISSN: 0955-2219

Peng J, Huang C, Cao C, et al., 2020, Inverse Nacre-like Epoxy-Graphene Layered Nanocomposites with Integration of High Toughness and Self-Monitoring, *MATTER*, Vol: 2, Pages: 220-232, ISSN: 2590-2393

Pelissari PIBGB, Pandolfelli VC, Carnelli D, et al., 2020, Refractory interphase and its role on the mechanical properties of boron containing nacre-like ceramic, JOURNAL OF THE EUROPEAN CERAMIC SOCIETY, Vol: 40, Pages: 165-172, ISSN: 0955-2219



## **Outreach**

### **CASC Industry Day**

The industry will take place virtually this year due to COVID. It is scheduled for the 19<sup>th</sup> of March 2021 and will have speakers from industry and from CASC presenting their latest results.

Industry speakers: Dr. Amanda Quadling (Director of Materials UK, Atomic Energy Authority), Dr. Adam Stevenson (R&D Manager for Engineered Ceramics North America, Saint Gobain)

CASC speakers: Dr. Samuel Humphry-Baker, Dr. Siyang Wang, Jack Lyons.

### **CASC Summer School**

The CASC Summer school was postponed to a later date due to COVID contact and travel restriction at that time.

### **Conferences**

Most conferences were postponed this year due to the pandemic.