

Centre for Advanced Structural
Ceramics
2021 Annual Report

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 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

Eduardo Saiz is the Director of CASC and he manages the centre in collaboration with the 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, 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.

The consortium has three levels of membership with a graduated annual fee and access to CASC facilities, people and projects (table belows).

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.

If you are interested in becoming a member of the CASC Industry Consortium, contact: Eduardo Saiz < e.saiz@imperial.ac.uk – 020 7594 6779>

	Diamond	Sapphire	Ruby
Type of membership	Strategic	Research & Training	Research & Training
Steering Group member	Yes	Yes	Yes
Equipment use: Free allowance up to Preferential rates	£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.

Professor 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.

Professor 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

Dr. Samuel Humphry-Baker	Imperial College Research Fellow
---------------------------------	---

Researchers

Dr. Iuliia Elizarova	Research Associate
Dr. Oriol Gavaldà Diaz	Research Associate
Dr. Siyang Wang	Research Associate
Dr. Madeleine Watson	Research Associate

PhD Students

Mercedes Baxter	2 nd year
Max Bennett	1 st year
Aaron Chote	2 nd year
Hugh Collett	2 nd year
Ben Currie	3 rd year
James Davidson	2 nd year
Lukas Egloff	1 st year
Max Emmanuel	3 rd year
Yinglung Hong	2 nd year
Tianhui Jiang	2 nd year
Ming Li	2 nd year
Jack Lyons	3 rd year
Le Mu	1 st year
Ollie Osborn	2 nd year
Harry Payne	2 nd year
Victoria Vilchez	2 nd year
William Wei	2 nd year
Kathryn Yates	3 rd year
Shitong Zhou	3 rd year
Muhammed Maktari	3 rd year



Research

Research Fellow Projects

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 NO_x 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.

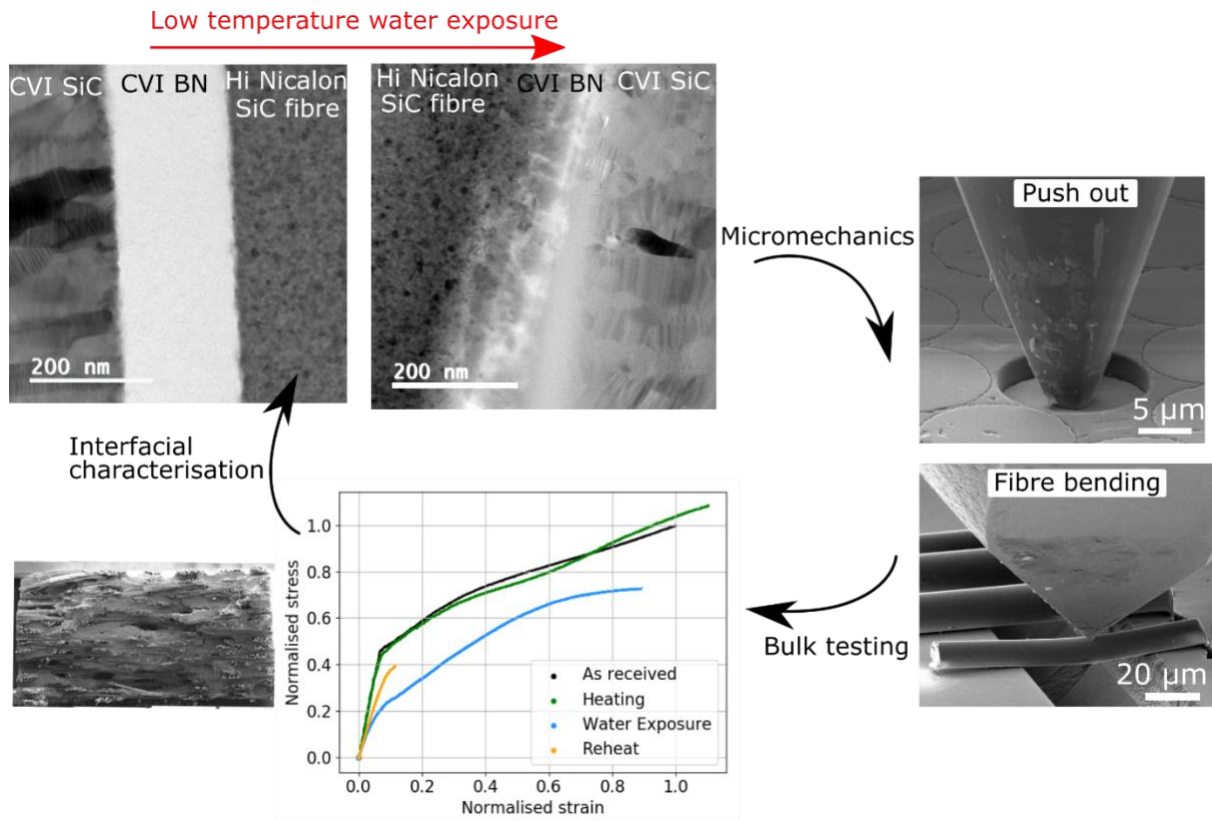


Figure 1 Schematic showing the methodology used to characterise the degradation of CMCs: (i) STEM analysis to understand the change in morphology and chemistry at the interfacial region, (ii) micromechanics of the interface and fibres and (iii) fractography after tensile bulk testing.

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 CO₂ 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 3-5um particles of the UHTC ZrB₂ 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 ZrB₂. 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

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 wastefrom 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)₂. 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.

Max Bennett

Fabrication of ceramic/ceramic metamaterials with deformable interphase

Interlocking mechanisms are in theory extremely effective at diffusing damages because it allows elements to slide but at the same time creates local crack-blocking compressive stresses in response to macroscopic crack-opening tensile stresses. The key objective of this thesis is to design an interfacial material to bind brittle micron-sized elements that provide toughening mechanisms at the nanometre scale. To develop any toughening mechanisms, the interfacial binder will have to break before the elements but then developed toughening mechanisms. In a first stage we will select an oxide-based composition, for its compatibility with the element composition, that could potentially deform plastically due to the high stresses level generated in between the elements at high temperatures. The material fracture will have to be measured in different crack opening modes to understand the link between interfacial material morphology, crystal plasticity, and element sliding.



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.

Lukas Egloff

Interlock-toughened metamaterials made with high resolution DLP printing

Interlocking mechanisms are in theory extremely effective at diffusing damages because it allows elements to slide but at the same time creates local crack-blocking compressive stresses in response to macroscopic crack-opening tensile stresses. Now the real challenge is to develop processes capable of programming interlocking in the microstructure at the micron and nano scale independently of the composition. The objective of this thesis is to develop a new process based additive manufacturing that will make possible the fabrication of interlocking elements at the micron scale in centimetre-sized sample. HD DLP can produce 3D sample by illuminating photo curable polymer layers through pixel based light projection. The fabrication of interlocking elements can thus be directly controlled using pixel-based exposure modulation in the layer produced.

Max Emmanuel

Study of grain boundary character and strength in WC-Co

Sponsor: Seco Tools

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 μm) 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 an emulsion-based approach to the fabrication of ceramic composites with controlled architectures. My analysis is based now in a model $\text{Al}_2\text{O}_3/\text{ZrO}_2$ and $\text{Al}_2\text{O}_3/\text{Metal}$ systems. We are study the rheological properties of emulsions containing ceramic particles and their dependence with their formulation. The mechanical properties of the materials obtained will be related to their microstructure.

Ming Li
Sponsor: President Scholarship

Development of new processing techniques for the fabrication of ceramic-based materials. I particular two areas: super-hydrophobic / oleophobic surface and self-healing 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
Sponsor: MTC

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.

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.

Kathryn Yates

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 α -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.

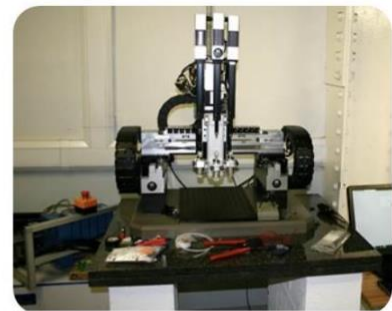
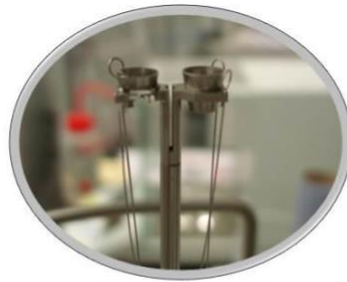
Shitong Zhou

Embedded 3D printing Multimaterial composites on ceramics

Sponsor: Imperial-CSC (Chinese Scholarship Council)

The project is to investigate embedded 3D printing multimaterial composites on ceramics, especially printing fibres 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 fibres and fills the voids between fibre and matrix. As an initial proof-of-concept, steel fibres are printed in Alumina slurry and the effect of rheological behaviour 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 analysed and tailored to improve the interface between fibres 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



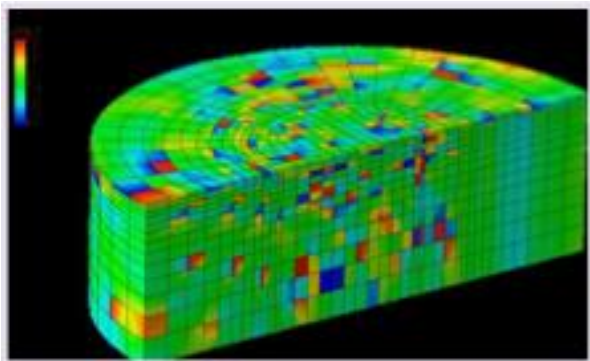
Although the purchasing and installation of large pieces of equipment by the Centre using the funding from the original CASC project is now completed, we continue to improve our experimental capability in this area using funds from other sources.

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, 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 B₄C and high temperature annealing of TiAlN, thermal treatments of high alumina castable refractories and producing composites of B₄C and SiC.

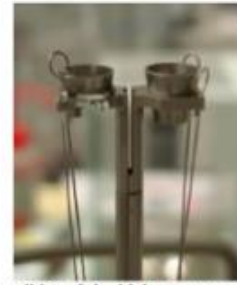
Thermal analysis

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.



Crucibles of the high temperature
combined TGA DTA

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 ZrB₂ and Al₂O₃ 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.

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).

Spark Plasma Sintering (SPS) or Field-Assisted Sintering Technology (FAST) furnace



The Spark Plasma Sintering (SPS) or Field-Assisted Sintering Technology (FAST) furnace is being installed and should be fully operational in January 2022. The furnace operates under inert atmosphere or vacuum at temperature up to 2200°C under a maximum of 100 kN of uniaxial pressure with heating and cooling rate routinely of 100°C/min, with a maximum of up to 500K/min.

The temperature is controlled using an axial pyrometer targeting the sample in the graphite die as well as several thermocouples to ensure the chamber and pistons stay within a safe temperature range during operation. The control and heating protocols are fully automated.

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 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. The CASC also purchased a new DLP system for high resolution printing, an ASIGA MAX X with maximum lateral resolution of 15 μm and height control down to 1 μm .

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.

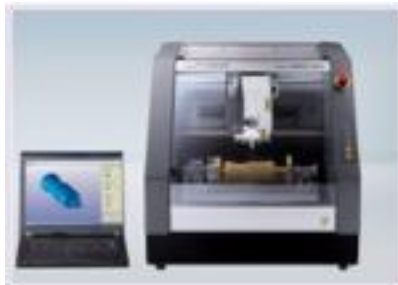


It is a three-part system: Extruder with Take Up, and Chopper Accessories. Maximum temperature 400°C and Variable speed control, 5 to 260rpm.

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



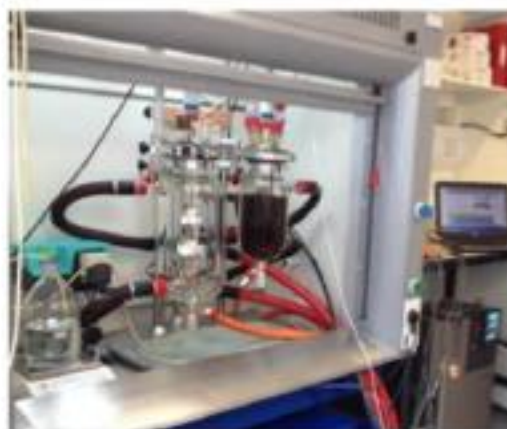
An Oxygen-Nitrogen-Hydrogen Elemental Mass Gas Analyser (Horiba, EMGA – 830 series) was installed at CASC in 2015. This includes also the Carbon-Sulphur Elemental Mass Induction Analyser (EMIA series) and a Glow Discharge-Optical Emission Spectroscopy (GD-OES) setup.

Graphene reactor

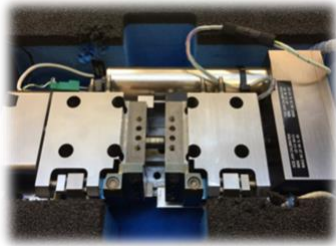
A one of a kind modular system for large- scale synthesis of chemically modified graphene, based on chemical graphite exfoliation, is in use. This system is flexible and allows for "on demand" fabrication of materials with tailored properties.

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

M Li, W Li, Q Guan, X Dai, J Lv, Z Xia, WJ Ong, E Saiz, X Hou. A Tough Reversible Biomimetic Transparent Adhesive Tape with Pressure-Sensitive and Wet-Cleaning Properties. *ACS nano* 15 (12), 19194-19201.

M Li, C Li, BRK Blackman, E Saiz. Energy conversion based on bio-inspired superwetting interfaces. *Matter* 4 (11), 3400-3414

O Gavalda-Diaz, R Manno, A Melro, G Allegri, SR Hallett, L Vandeperre, ... Mode I and Mode II interfacial fracture energy of SiC/BN/SiC CMCs. *Acta Materialia* 215, 117125

C Li, M Li, Z Ni, Q Guan, BRK Blackman, E Saiz. Stimuli-responsive surfaces for switchable wettability and adhesion. *Journal of the Royal Society Interface* 18 (179), 20210162

CLA Leung, I Elizarova, M Isaacs, S Marathe, E Saiz, PD Lee. Enhanced near-infrared absorption for laser powder bed fusion using reduced graphene oxide. *Applied Materials Today* 23, 101009

O Gavalda-Diaz, J Lyons, S Wang, M Emmanuel, K Marquardt, E Saiz, ... Basal Plane Delamination Energy Measurement in a Ti_3SiC_2 MAX Phase. *JOM*, 1-7

I Azcune, A Huegun, A Ruiz de Luzuriaga, E Saiz, A Rekondo. The effect of matrix on shape properties of aromatic disulfide based epoxy vitrimers. *European Polymer Journal* 148, 110362

RMG De Meyere, K Song, L Gale, S Harris, IM Edmonds, TJ Marrow, ... A novel trench fibre push-out method to evaluate interfacial failure in long fibre composites. *Journal of Materials Research*, 1-10

Aldegaither N, Sernicola G, Mesgarnejad A, Karma A, Balint D, Wang J, Saiz E, Shefelbine SJ, Porter AE, Giuliani F, 2021, Fracture toughness of bone at the microscale, *Acta Biomaterialia*, Vol: 121, Pages: 475-483
M Li, C Li, BRK Black, E Saiz. Mimicking

nature to control bio-material surface wetting and adhesion. *International Materials Reviews*

C Cao, Z Lin, X Liu, Y Jia, E Saiz, SE Wolf, HD Wagner, L Jiang, Q Cheng. Strong Reduced Graphene Oxide Coated Bombyx mori Silk. *Advanced Functional Materials*, 2102923

C Cao, J Peng, X Liang, E Saiz, SE Wolf, HD Wagner, L Jiang, Q Cheng. Strong, conductive aramid fiber functionalized by graphene. *Composites Part A: Applied Science and Manufacturing* 140, 106161

Wang S, Lovell E, Guo L, Wilson N, Ryan MP, Giuliani F, 2021, Environment-assisted crack nucleation in La(Fe,Mn,Si)13-based magnetocaloric materials, *International Journal of Refrigeration*

Vreeswijk M, Kot A, Giuliani F, Humphry-Baker S, 2021, Abnormal WC crystal growth from liquid Co flux occurs via eta phase decomposition, *International Journal of Refractory Metals and Hard Materials*

Gavalda-Diaz O, Manno R, Melro A, Allegri G, Hallett SR, Vandeperre L, Saiz E, Giuliani F, 2021, Mode I and Mode II interfacial fracture energy of SiC/BN/SiC CMCs, *Acta Materialia*, Vol: 215, Pages: 1-11

De Meyere RMG, Song K, Gale L, Harris S, Edmonds IM, Marrow TJ, Saiz E, Giuliani F, Armstrong DEJ, Gavalda-Diaz O, 2021, A novel trench fibre push-out method to evaluate interfacial failure in long fibre composites, *Journal of Materials Research*, Vol: 36, Pages: 2305-2314

Emmanuel M, Gavalda-Diaz O, Sernicola G, Msaoubi R, Persson T, Norgren S, Marquardt K, Britton TB, Giuliani F, 2021, Fracture energy measurement of prismatic plane and $\Sigma 2$ boundary in cemented carbide, *JOM*, Vol: 73, Pages: 1589-1596

Xu Y, Gu T, Xian J, Giuliani F, Britton T, Gourlay C, Dunne F, 2021, Intermetallic size and morphology effects on creep rate of Sn-3Ag-0.5Cu solder, *International Journal of Plasticity*, Vol: 137

Aberdeen S, Hur CA, Cali E, Vandeperre L, Ryan MP, 2022, Acid resistant functionalised magnetic nanoparticles for radionuclide and heavy metal adsorption, *Journal of Colloid and Interface Science*, Vol: 608, Pages: 1728-1738

Ewenz Rocher M, Hermann T, McGilvray M, Ifti HS, Vieira J, Hambidge C, Quinn MK, Grossman M, Vandeperre L, 2022, Pressure-sensitive paint diagnostic to measure species concentration on transpiration-cooled walls, *EXPERIMENTS IN FLUIDS*, Vol: 63

Humphry-Baker S, Vandeperre L, 2021, Creep deformation of WC hardmetals with iron-based binders, *International Journal of Refractory Metals and Hard Materials*, Vol: 95, Pages: 1-8

Collett H, Bouville F, Giuliani F, Schofield E, 2021, Structural Monitoring of a Large Archaeological Wooden Structure in Real Time, Post PEG Treatment, *FORESTS*, Vol: 12

Magrini T, Kiebala D, Grimm D, Nelson A, Schrettl S, Bouville F, Weder C, Studart AR, 2021, Tough Bioinspired Composites That Self-Report Damage, ACS APPLIED MATERIALS & INTERFACES, Vol: 13, Pages: 27481-27490

Poloni E, Bouville F, Dreimol CH, Niebel TP, Weber T, Biedermann AR, Hirt AM, Studart AR, 2021, Tough metal-ceramic composites with multifunctional nacre-like architecture, SCIENTIFIC REPORTS, Vol: 11

T Magrini, F Bouville, AR Studart. Transparent Materials with Stiff and Tough Hierarchical Structures. Open Ceramics, 100109

Beyer C, Myhill R, Marquardt K, McCammon CA, 2021, A reversed redox gradient in Earth's mantle transition zone, EARTH AND PLANETARY SCIENCE LETTERS, Vol: 575

Gardes E, Gibouin D, Radiguet B, David A, Prellier W, Marquardt K, 2021, Magnesium transport in olivine mantle: new insights from miniaturized study of volume and grain boundary diffusion in Mg₂SiO₄ bi-crystals, CONTRIBUTIONS TO MINERALOGY AND PETROLOGY, Vol: 176

Ferreira F, Hansen LN, Marquardt K, 2021, The Effect of Grain Boundaries on Plastic Deformation of Olivine, JOURNAL OF GEOPHYSICAL RESEARCH-SOLID EARTH, Vol: 126

Elazar O, Kessel R, Huang J-X, Marquardt K, Navon O, 2021, Silicic microinclusions in a metasomatized eclogite from Roberts Victor mine, South Africa, LITHOS, Vol: 388

Avadani D, Hansen L, Darnbrough E, Marquardt K, Armstrong D, Wilkinson A, Micromechanical testing of olivine grain boundaries

Bollinger C, Ferreira F, Marquardt K, Intragranular plasticity vs. grain boundary sliding in forsterite: microstructural evidence, Geochemistry, Geophysics, Geosystems

Marquardt K, Dobson D, Hunt S, Faul U, Grain boundary character information via individual imaging or statistical analyses: complexion transitions and grain boundary segregation



Outreach

CASC Industry Day

The industry took place virtually this year due to COVID the 19th of March 2021 and had 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.