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Engineering and Physical Sciences Research Council

EPSRC Programme Grant:

Enabling Next Generation Additive Manufacturing

Annual Report 2020

UNIVERSITY OF BIRMINGHAM

WARWICK

Contents

Front Cover: Conductive and translucent polymer, PEDOT:PSS (coloured squares), printed by Dimatix inkjet using in-house ink formulations, deposited on inkjet printed silver traces designed for rapid 4-wire sheet resistance measurements.

Strain sensor printed on a stretchable substrate

Overview

This EPSRC Programme Grant's vision is to develop next generation Additive Manufacturing (AM) to enable the manufacture of whole systems rather than component parts. We therefore focus on understanding how to control the co-deposition (interfacing) of both functional and structural materials to significantly extend AM's reach beyond the well understood geometric freedoms they currently enable.

The Programme centres on the exemplar application areas of 3D electronics, pharmaceuticals, healthcare devices and regenerative medicine, and is organised into four Research Challenges (RCs). RC1 is concerned with developing key analytical methods to determine interface evolution between deposited materials; RC2 is establishing a predictive computational framework to optimise deposition strategy; RC3 works to overcome anisotropy in deposited functional materials, and RC4 looks to achieve the controlled co-deposition of multimaterials. There is considerable overlap between the RCs which is starting to produce exciting, high impact results. Notable advances have been made using the University of Nottingham's new OrbiSIMs system, which we are able to exploit to map, with high chemical specificity and spatial precision, the hybrid organic/inorganic interfaces in 3D printed flexible electronics devices. This has given us, for example, key insights into the role of organic residues in metal nanoparticle inks. We have also extended the range of processes available to the Programme through the acquisition of a new Projection Micro Stereolithography (PμSLA) system, which we are adapting for multimaterial processing.

Our Programme spans many disciplines ranging from engineering, physics and mathematics to chemistry and biology. It also spans three universities – Nottingham, Warwick and Birmingham, and comprises a team of highly skilled researchers and PhD students. These researchers are supported by technical/administrative colleagues and is led by a committed investigator team. This year we bade farewell to post-doctoral researcher, Dr Qin Hu, and welcomed Dr Lea Santu, alongside three new doctoral students (Nur Rofiqoh Eviana Putri, Tien Thuy Quach and Jonathan Austin). We have also continued to successfully collaborate with high profile academic and industrial partners such as NPL (UK), Karlsruhe Institute of Technology (KIT, Germany), Canon Production Printing (Netherlands), AWE (UK), and the Lawrence Livermore National Laboratory (USA). We continue to be grateful for the support of our funder the EPSRC and the valued contribution of our Advisory Board.

Of course, our Programme has, like everyone, had a disrupted year where because of the Covid-19 pandemic our laboratories closed for several months due to the necessity to work from home. However, this time was productively used to analyse data, submit funding applications, prepare journal papers for publication in high impact journals and plan our future experimentation. Thanks to the hard work of colleagues (led by Senior Research Technician, Mark East), our laboratories were amongst the first at Nottingham to re-open in the summer, adopting new practises to ensure that we work in a Covid-safe manner. Since the summer, we have increasingly resumed laboratory research work and overall, we have managed to make significant progress during year three of our Programme.

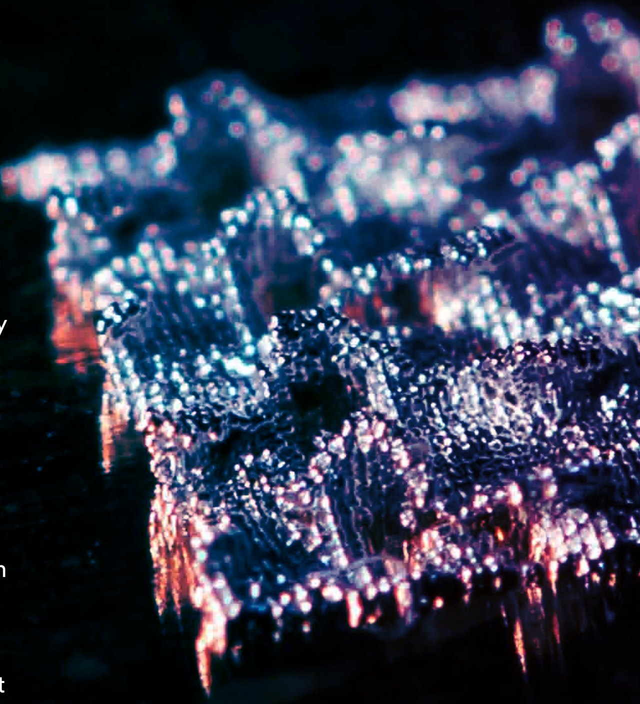
Having just passed the half-way point of the grant, we were pleased to have submitted our mid-term review (MTR) to the EPSRC in July 2020. The MTR gave us an opportunity to reflect on the work undertaken thus far and plan our future activity so as to maximise the impact of our Programme. I am happy to report that the reviewers' and panel's comments were overwhelmingly positive and we were awarded an overall score of 9/10 (Very Strong) – a recognition of the exemplary standards that the Programme is working to.

As we are effectively trying to overcome the challenges of creating 21st century manufacturing tools that are agnostic to what they make, we need to thoroughly examine these with exemplars to test our research outcomes. Thus, now that we have passed the half-way point of the Programme, in order to drive the research to the next level whilst maximising the accessibility of our work, we have reversed the emphasis of the activity from being entirely RC-led (with applications as exemplars) to being increasingly exemplar led (where RCs are focused at overcoming the challenges of how to make multifunctional AM devices in our application areas). In many respects, this is a subtle change, but one that increases partner engagement and take up, and gives clear focus. Additionally, it will help us provide the evidence we need to execute our publication strategy by turning good quality 3* publications into excellent 4* papers due to the tangibility of the associated applications.

Entering our fourth year of research, the Programme will continue to push back boundaries and communicate our work to wider audiences. We will continue to publish our work and have an ever-increasing list of high impact journal publications. When possible, we will also resume research visits, exchanges and engagement activities which were paused in 2020 due to the pandemic. In particular, we have been invited to present as part of the prestigious Royal Society Summer Science Exhibition 2021, which was postponed in 2020 and will be via a digital platform this time.

I hope you enjoy reading this report which outlines how the Programme has started to deliver its vital contribution toward achieving Next Generation Additive Manufacturing.

Professor Richard Hague
Principal Investigator



Demonstrating the capability of MetalJet to print gyroid structures of tin onto a copper substrate

Technical highlights of year three

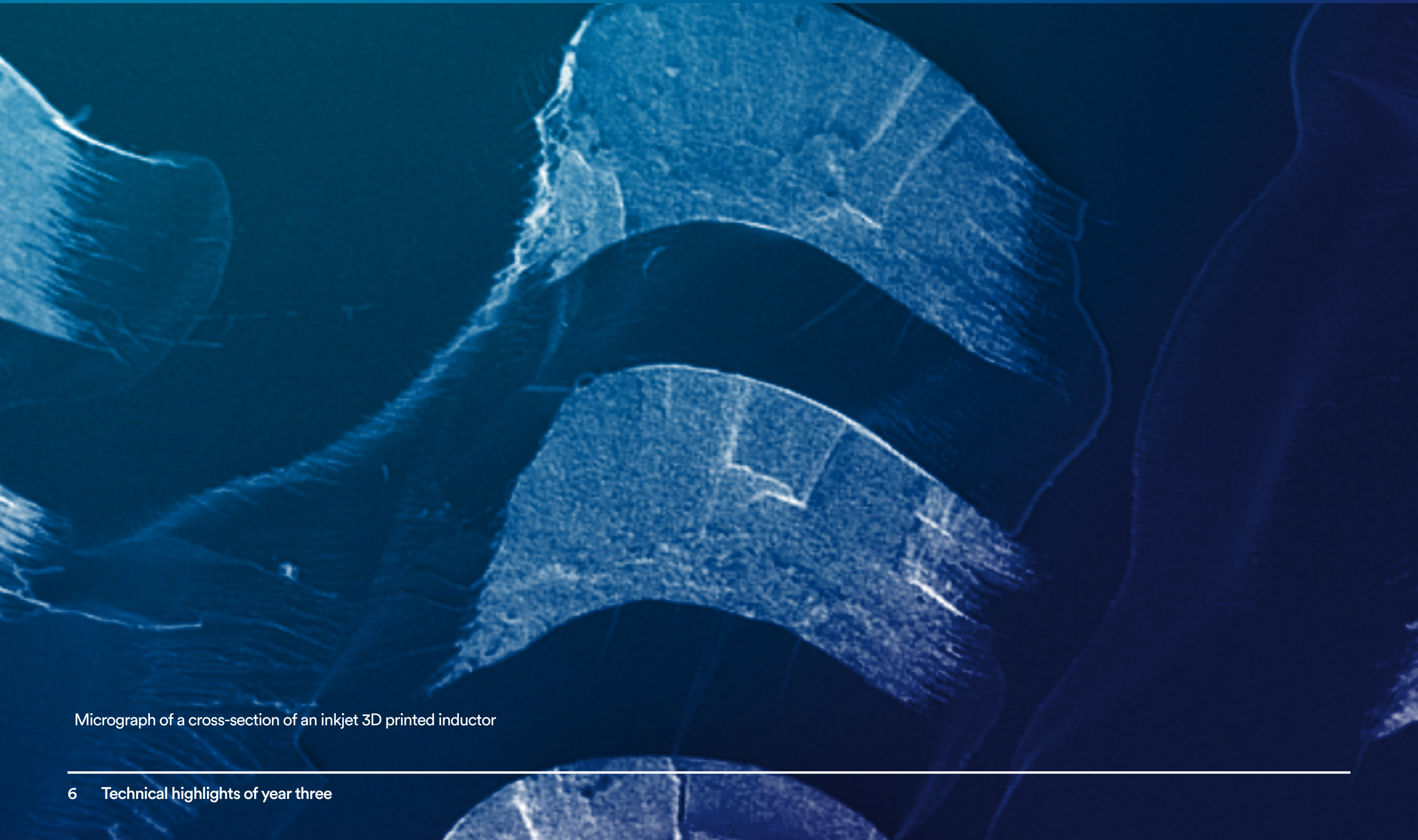
There have been a great many highlights this year including a flurry of publications in leading journals, bringing the total number to 18.

A further five papers have been submitted and there are an impressive 38 being prepared. We were also delighted that the paper by He *et al* [Adv Ther 2020] featured on the front cover of the journal *Advanced Therapeutics* – a first for our Programme.

We present a selection of our notable achievements during year three:

Analytical methods

- Used the novel 3D OrbiSIMS in combination with other analytical methods to reveal that anisotropic electrical conductivity of inkjet printed metal nanoparticles is caused by organic residuals from metal nanoparticle inks
- Tailored the novel 3D OrbiSIMS operation for the analysis of complex samples produced across all RCs with multiple research outputs
- Developed a protocol for the measurement of the degree of vinyl group consumption in photo-curable, acrylate-based inkjet printed samples. The results have been used to validate a physics model developed by the RC2 team, which enabled the creation, by the RC3 team, of a platform for designing optimal and cost-effective printing strategies
- Commenced a new externally funded project to apply characterisation methodologies and explore multimaterial additive manufacturing routes for producing Micro-Patterned Gaseous Detectors



Micrograph of a cross-section of an inkjet 3D printed inductor

Processes

- Set up a new Projection Micro Stereolithography (PμSLA) process and started building a multi-material printing unit for manufacturing high precision multifunctional devices
- Functional formulations (giving controlled drug release or bacterial biofilm resistance) have been developed for both material jetting and PμSLA
- Manufactured an oral dosage form tablet with a controlled drug distribution and release-profile by using a new pro-drug formulation strategy and multimaterial jetting technique
- In collaboration with Added Scientific Ltd, started building a multimaterial jetting system for printing high viscosity ink formulations
- New high-temperature substrate heating has been shown to greatly improve inter-droplet bonding in high-temperature metals, achieving better consolidation in structures printed using MetalJet
- Influence of different MetalJet process parameters on the quality of inter-droplet and droplet-to-substrate bonding were further examined through an integrated experimental and computational approach
- Optimised the two photon polymerisation (2PP) deposition process for a library of polymeric materials to achieve stable 3D structures
- Demonstrated successful 2PP fabrication of multimaterial structures suitable for a range of applications from electronic devices to drug delivery
- 2PP printed structures were demonstrated on flexible substrates, which enables their exploitation for flexible electronics
- 2PP printed structures were successfully lifted off glass substrates and transferred onto a different substrate and/or into solution, with no loss of shape/geometry. This opens up the exciting prospect for the integration of these structures with materials or devices produced by different methods. The ability to suspend these structures in solution is of key importance for their use in e.g. drug delivery
- Experimentally examined the effect of printing parameters on the resulting properties of 2PP structures, thus providing a basis for the development of a theoretical model
- Developed post-deposition functionalisation of 2PP structures with gold nanoparticles (AuNP)

Modelling

- Proposed a novel theoretical, descriptive and predictive model for inkjet printing to aid the understanding of the fundamental physical mechanisms, accurately predict the outcome of real processes, and identify the best possible component designs and manufacturing processes for specific applications
- Developed a computational framework for the spreading of droplets over solid surfaces, capturing critical small-scale physics associated with the liquid-solid interactions, that will form the basis of advanced observation-driven computational models for the complex MetalJet deposition process
- Developed and applied a finite element model for drop cooling, coupled to the underlying solid mechanics, to understand detrimental experimentally-observed effects in MetalJet deposition
- Carried out a thorough study of an existing model of 2PP and extended it by including explicit space dependence

Functional materials and demonstrators

- Improved hyperbranched (HB) polymer synthesis giving a wider library of available materials for AM
- Formulated reliable PEDOT:PSS inks suitable for fully-printed LED fabrication, and as printed transparent electrochemical anodes for bio-photovoltaic cells
- Provided insight into the quantum transport phenomena as well as the structural properties of inkjet printed 2D materials
- For the first time, successfully replaced single-layer graphene with inkjet printed graphene as a contact material for 2D metal chalcogenides
- Developed a new thiol-based conductive gold ink formulation which enhances the cohesion of printed layers and thus provides structural integrity for printed electrodes in 2D & 3D
- Demonstrated the feasibility of an inkjet printed drug release monitoring RF sensor for a transdermal drug delivery skin patch
- The production of alloys *in situ*, using Metaljet printheads was showcased, opening up opportunities for new materials in droplet-on-demand metal jetting
- Developed a photo-curable biodegradable bio-ink that supports cell adhesion and proliferation which can be printed using PμSLA and achieve a 10μm resolution

Key individuals

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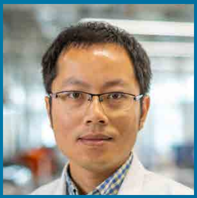

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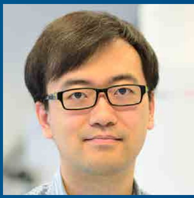

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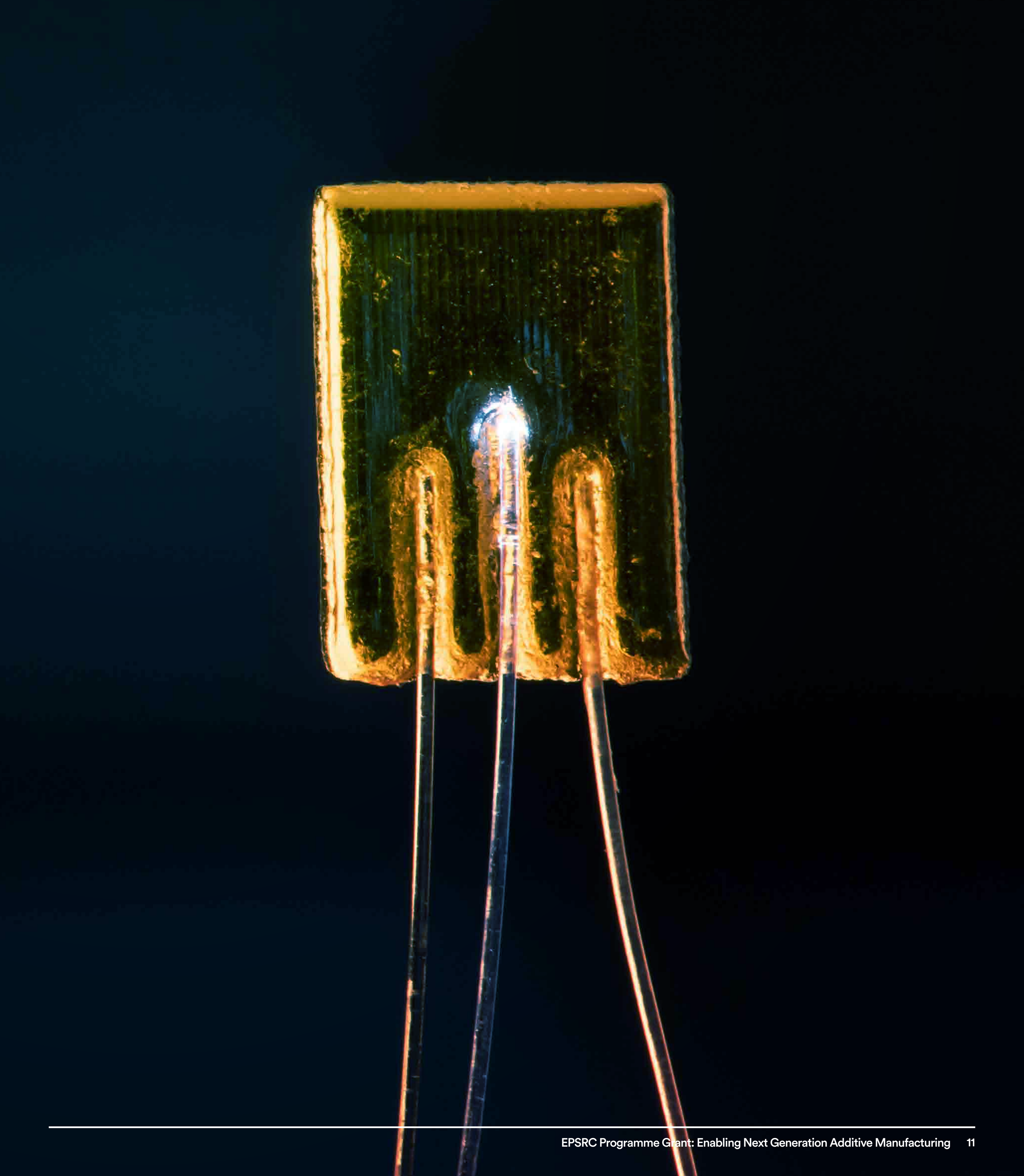


Inkjet printed health patch for blood oxygen saturation detection, using transparent TPGDA (Tripropylene glycol diacrylate) polymers

Research challenge 1 (RC1)

Determination of the interface/interphase evolution at the microscale

Aim: *To understand, through precision experiments, the spatio-temporal interface/ interphase evolution between successively deposited droplets or voxels of multimaterials at the microscale*

Our primary challenge lies in the inter and intra layer coalescence/ bonding for functional-structural or functional-functional materials. This is due to differences in the materials' physical state, chemistry, and temperature at deposition or conversion. To overcome these, we have developed new methodologies for insightful *ex situ* materials analysis techniques. RC1 has emerged as an underpinning activity for the Programme, where in the first year, we established appropriate micro/nanoscale analytical methods for both the chemical and physical interrogation of interfaces within samples. These methods are now routinely integrated and implemented across the Programme to inform both modelling and build optimisation strategies, and include:

- a combination of focused ion beam scanning electron microscopy (FIB-SEM) and time-of-flight secondary ion mass spectrometry (ToF-SIMS) to expose and chemically analyse with high chemical specificity and spatial precision the hybrid organic/inorganic interfaces in 3D printed flexible electronics devices, developed in collaboration with NPL
- a FIB-SEM methodology for exposing the interface between MetalJet droplets, where a Ga+ ion beam both controllably removes the upper section of neighbouring droplets and then images the grain structure shared across droplet interfaces to assess their metallurgical bonding. Integrated with other analytical techniques (i.e. AFM, SIMS), this provides a rich seam of data and understanding to inform the modelling and optimisation of the printing procedure
- a protocol for the systematic measurement of the relative degree of conversion from monomer to polymer in photo curable, acrylate-based inkjet printed structures using confocal Raman microscopy, with subsequent data analysis to spatially track the degree of vinyl group consumption. Combined with RC2, this has given clear insights into the temporal and spatial deposition strategies required for consistent curing
- a combination of electrical resistivity tests, morphological analysis and novel 3D nanoscale chemical analysis of printed devices using silver nanoparticles (AgNPs), which we employed to show for the first time that the polymer stabiliser polyvinylpyrrolidone (PVP) tends to concentrate between vertically stacked AgNP layers, as well as at dielectric/conductive interfaces

Together with NPL, we are also developing new methods to apply a state of the art hybrid time-of-flight/orbitrap secondary ion mass spectrometry (3D OrbiSIMS) instrument, which allows label-free 3D chemical imaging of materials, cells and tissues with exceptionally high mass resolution and chemical specificity. The primary advantage of this instrument is the ability to provide great depth resolution alongside outstanding mass resolution. We are fortunate that, though this work was originally scheduled to be conducted at NPL, Nottingham have been successful in acquiring its own 3D OrbiSIMS through a strategic equipment grant and thus we now have considerably more access than originally envisaged. Since the arrival of the instrument in February 2019, we have tailored its operation for the analysis of complex organic/inorganic samples produced across all the processes – for example: a 3D inkjet printed pharmaceutical implant where we were able to unambiguously determine the distribution of drugs and polymers; and multimaterial 2PP microstructures, where we processed high resolution mapping data with machine learning to spatially identify areas that we selectively functionalised with gold nanoparticles. We will continue to explore this novel instrument and will exploit its potential for the remainder of the Programme.

The expertise developed in the Programme has helped us to obtain a research grant of £40,000 funded by FAPESP and the University of Nottingham, in collaboration with the University of Sao Paulo (Brazil) and CERN, to apply the characterisation methodologies and explore multimaterial additive manufacturing routes for producing Micro-Patterned Gaseous Detectors used in nuclear and particle physics.

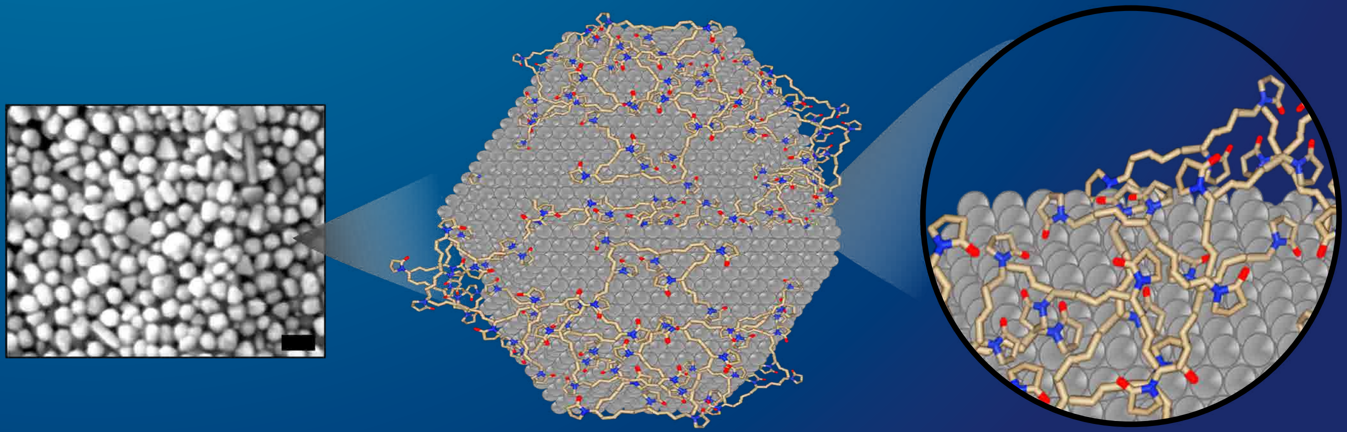
3D OrbiSIMS results showing surface chemistry mapping of polymer microspheres fabricated with microfluidics for 3D printing applications

Residual polymer stabiliser causes anisotropic conductivity in metal nanoparticle 2D and 3D printed electronics

Inkjet printing of metal nanoparticles (MNPs) allows for design flexibility, rapid processing and enables 3D printing of functional electronic devices through co-deposition of multiple materials. However, the performance of printed devices, especially their conductivity, is lower than those made by traditional manufacturing methods and was previously not fully understood. In year three, we concluded a piece of research that revealed that anisotropic electrical conductivity of printed MNPs is caused by organic residuals from MNPs inks. We employed a combination of electrical resistivity tests, morphological analysis and novel 3D nanoscale chemical analysis of printed devices using silver nanoparticles (AgNPs) to show that the polymer stabiliser polyvinylpyrrolidone (PVP) tends to concentrate between vertically stacked AgNP layers, as well as at dielectric/conductive interfaces. The understanding of organic residue behaviour in printed nanoparticles will reveal potential new strategies to improve nanomaterial ink formulations for functional printed electronics, which will be explored in the upcoming years in collaboration with RC3 and RC4. We will now employ the same characterisation methodology to investigate and drive strategies for novel ink formulations containing nanoscale materials such as graphene, hexagonal boron nitride and functionalised gold nanoparticles.

New developments in 3D micro/nano scale mass spectrometry

The University of Nottingham's 3D OrbiSIMS instrument is extremely relevant for the analysis of interfaces in multimaterial multifunctional additive manufacturing samples, as in most cases the materials will have a complex geometry at the micro/nano scale and are molecularly complex in order to carry out a function. Since the arrival of the instrument, we have tailored its operation for the analysis of complex samples produced across all RCs. Those include hybrid organic-inorganic materials as well as multimaterial printing for biomedical devices, multi-drug formulation printing for tailored delivery in implants, functional metal nanoparticles and optoelectronic devices. Many of the materials produced in the project will yield extremely complex 3D OrbiSIMS signals, therefore, to make full use of those signals and aid data interpretation/visualisation, statistical data analysis methodologies have been developed and tailored for multimaterial additive manufacturing samples. These include unsupervised machine learning approaches for dimensionality reduction. All methods are packaged with a new software, which is in continuous development. In the coming year, we will continue to develop and refine the operation of the 3D OrbiSIMS to further analyse and characterise multimaterial samples produced by the Programme.



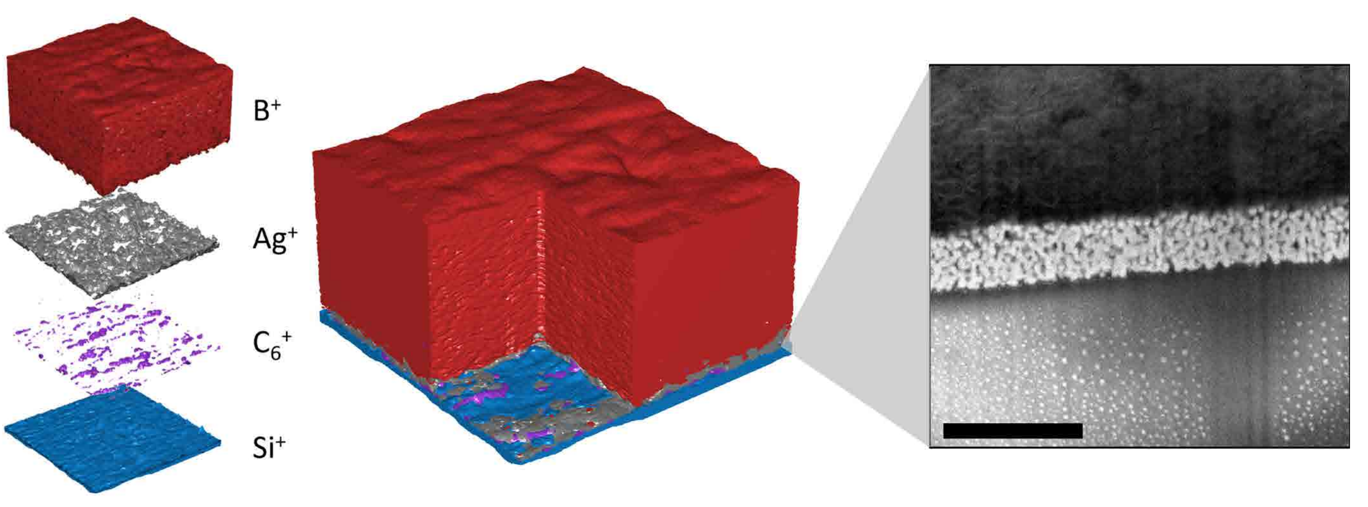
Micrograph of inkjet printed silver nanoparticles and schematic showing polymer stabiliser polyvinylpyrrolidone encapsulating individual nanoparticles. The scale bar represents 100 nm

Methodologies for surface, interface and interphase analysis in multimaterial, multi-functional additive manufacturing

We have developed methodologies for *ex situ* materials analysis techniques capable of delivering complete 3D characterisation of samples with chemical specificity and high resolution at the micro/nano scale. These core analytical techniques span a wide range of analytical spot sizes (spatial resolution) and chemical specificity, and include: scanning and transmission electron microscopy (SEM & TEM), focused ion beam scanning electron microscopy (FIB-SEM), atomic force microscopy (AFM), optical profilometry, X-Ray photoelectron spectroscopy (XPS), time-of-flight and orbitrap secondary ion mass spectrometry (ToF-SIMS and OrbiSIMS), confocal Raman microscopy and ultra-microtomy. Some examples include a FIB-SEM methodology to expose and assess the interface between MetalJet droplets, which aided the modelling of the MetalJet process by the RC2 team and optimisation of the printing procedure. FIB-SEM has also been employed to measure an inkjet printed field effect transistor for photo sensing applications created by RC4 colleagues. We also developed a protocol for the systematic measurement, using confocal Raman microscopy, of the degree of vinyl group consumption in photo curable inkjet printed materials. The results have been used to validate a physical model developed by the RC2 team, which enabled the creation, by RC3 of a platform for designing optimal and cost effective printing strategies. We will continue to employ the established methods and create new ones for novel materials produced in the Programme.

3D printing of micro-patterned gaseous detectors

RC1 postdoctoral researcher, Gustavo Ferraz Trindade has been awarded a research grant of £40,000 funded by FAPESP for a collaborative project with the University of Sao Paulo (Brazil) and CERN, to apply characterisation methodologies and explore multimaterial additive manufacturing routes for producing Micro-Patterned Gaseous Detectors. Gas Electron Multiplier (GEM) detectors are used in nuclear and particle physics for radiation detection. They consist of layers of a dielectric material sandwiched by two layers of conductive material, thus having a great overlap with the hybrid organic/inorganic interfaces studied in our Programme. Currently the detectors used at CERN are produced by lithography and chemical etching. Such a method limits geometric variation of the devices and is relatively slow to produce modified designs. This new project has the potential to generate disruptive impact on High Energy Physics experiments and facilities that will benefit from novel/improved designs of GEMs. We will now also investigate possible methods of depositing metal layers, test the performance of 3D printed GEMs with alternative geometries and study interface degradation of used GEMs. For that, we need to employ the chemical interface characterisation developed in RC1.



3D chemical distribution and micrograph of a cross-section of an inkjet printed field effect transistor. The scale bar represents 1 µm

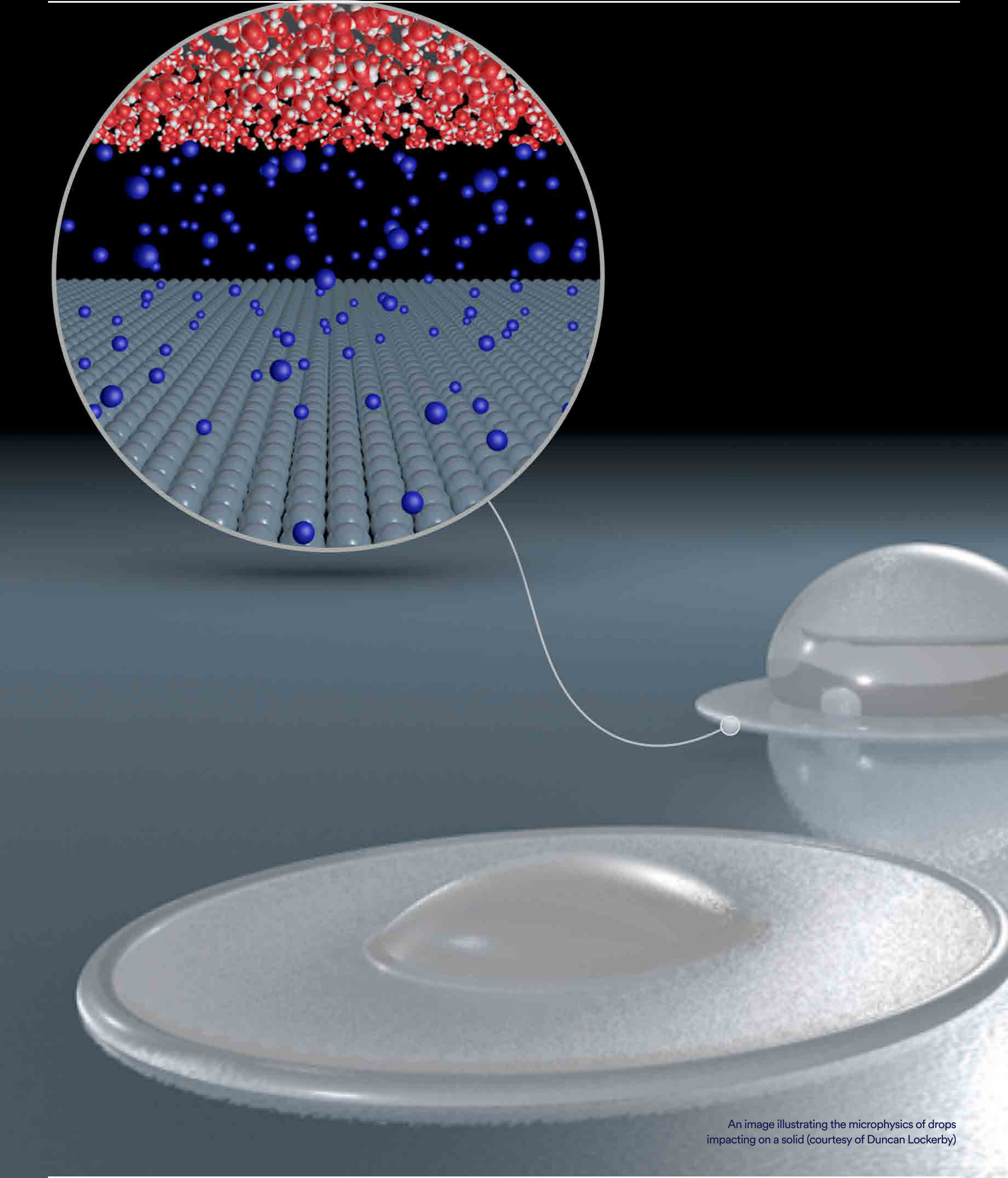
Research challenge 2 (RC2)

Multifunctional AM computational modelling framework

***Aim:** To accurately model and simulate multimaterial AM: from constituent materials to multifunctional components*

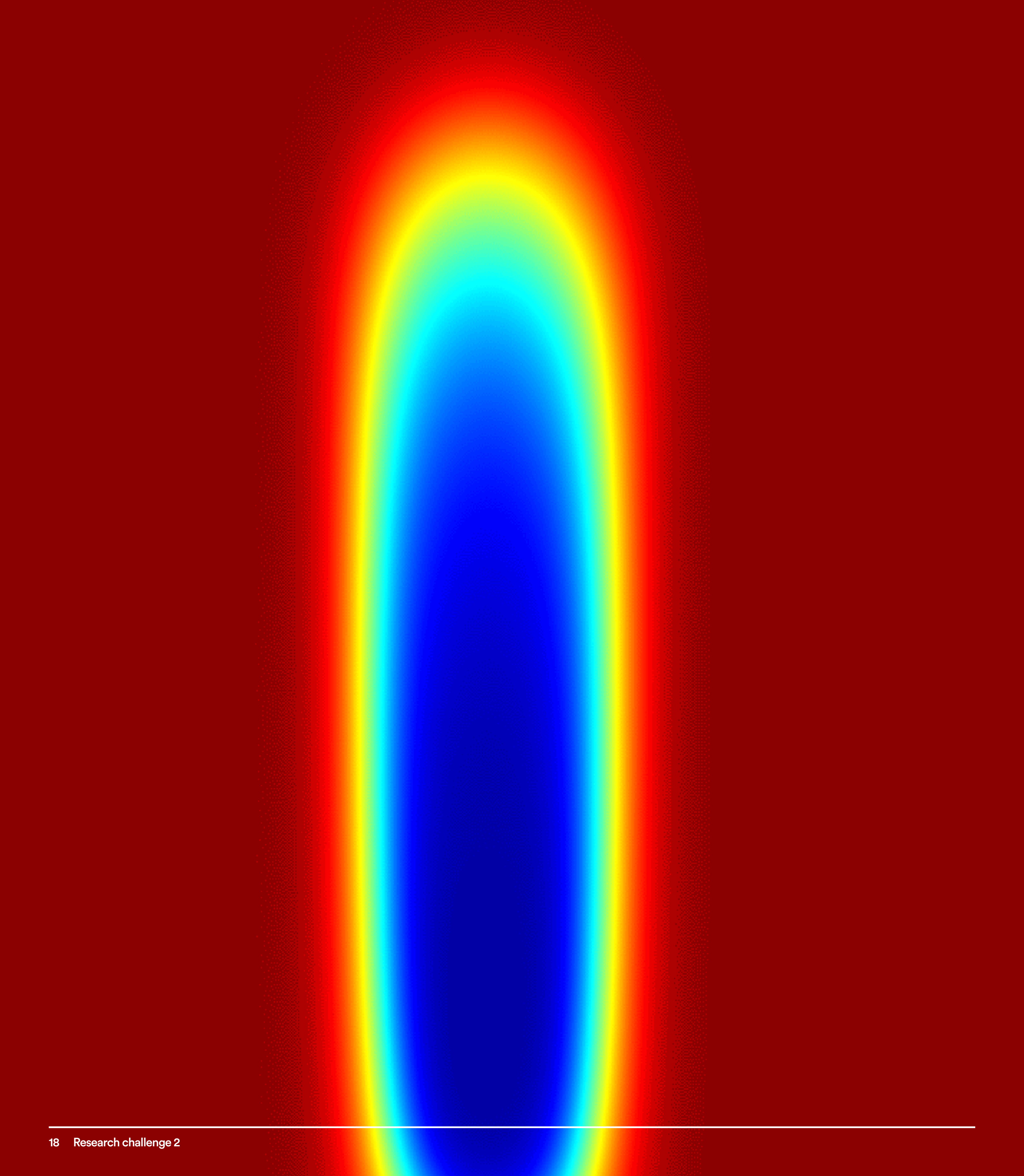
This research challenge is at the core of our tri-institutional cross-disciplinary collaboration, with the juxtaposition of theory, experimentation and application promising major breakthroughs in our understanding of a range of emerging multimaterial AM technologies. Computational models have now been developed, hand-in-hand with targeted experimental analysis, to (i) descriptively aid understanding of the fundamental physical mechanisms and supplement experimental work with targeted virtual experiments; (ii) predict, using validated models, the outcome of real processes (and the resultant properties of structures made) to rapidly explore process or design variables; and (iii) combine with optimisation algorithms to identify the best possible functional component designs and manufacturing processes for specific applications. Highlights so far from modelling the processes are:

- MetalJet: for microdrop flight dynamics, a new computationally verified formulae and associated web-app have been created to predict the critical level of drop cooling between ejection and deposition, with experimental validation currently underway. For the microdrop impact phase, an open-source volume-of-fluid framework has been extended and tailored to provide the first real time analysis of the interplay of fluid dynamics, heat transfer and solidification, combining in a complex nonlinear fashion to produce a final structure. For solidification, advanced FEA models can now be deployed. These progressions have both stimulated further theoretical investigations into the crucial dynamics of the three-phase contact line, and also stimulated new lines of synergistic experimental investigations between the Programme's theoreticians and experimentalists.
- Inkjet: where a novel methodology capable of prediction and optimisation of the spatial variation in UV curing has been developed by a multi-disciplinary team of experimentalists and modellers. A systematic descriptive and predictive model has been proposed to predict the degree of vinyl group consumption spatially by considering the critical processing parameters, including UV source pathway, intensity, voxel sizes and interlayer attenuation. The proposed and validated methodology has been successfully applied to improve printing performance and will be implemented within the aforementioned open-source volume-of-fluid CFD framework. This will provide a detailed prediction of the inkjet printing phenomena, including single and compound drop formation and coalescence, heat/mass transfer from liquid-gas interfaces, and polymer cure, enabling its use as a comprehensive virtual design tool for reactive inkjet.
- Two Photon Polymerisation (2PP): a process that has thus far lacked insight from theoretical analysis, has motivated us to initialise a novel project to address this deficiency. An existing model for a single voxel has been studied in detail and work is under way to extend it to a full structure-level model, with close collaboration between experimental chemists and theoreticians in the Programme. This will be validated against experimental data sets in a range of parameter regimes for the level of curing, to provide a predictive tool for the optimally-cured manufacture of complex structures.



An image illustrating the microphysics of drops impacting on a solid (courtesy of Duncan Lockerby)

Snapshot from a 2D simulation of a line being written using the 2PP process. Different colours correspond to different levels of polymerisation (dark blue is the highest level)



MetalJet - predictive modelling of dynamic wetting, heat transfer and phase transitions in additive manufacturing

Predicting the shape of the solidified droplet in drop-on-demand AM from the system parameters requires the use of mathematical models that incorporate the in-flight cooling, complex interaction between droplet deformation upon impact, dynamic wetting, heat transfer, phase transitions and solid mechanics during cooling. Using established black-box commercial software, accurate modelling is not possible for all aspects of the process. Hence we seek to produce a modelling platform that can include all the relevant physics and inform the development of AM processes such as MetalJet.

RC2 modellers have started to collaborate with experimentalists to validate our theoretical approach to calculating the extent of MetalJet droplet cooling in flight. Based on the first experimental results, work is currently under way to revise the proposed experimental procedure to make clear-cut conclusions. We have also been successful in implementing a mathematical model that can capture the interplay between droplet deformation, flow inside the droplet and dynamic wetting. This project will now implement obtuse contact angle mechanics for the early stages of wetting, followed by heat transfer, interface formation and phase-change effects. Finally, upon solidification, a finite element model has been developed to establish how cooling and solid deformation of single and multiple droplets can explain the apparent lack of adhesion at the interface in certain systems, and provide complementary understanding to experimental analysis.

A modelling framework for the inkjet-deposited UV-cured creation of polymeric materials

This project aims to develop a systematic descriptive and predictive model for inkjet printing that captures the relationship between drop-on-demand inkjet printing strategies, UV dose and ink polymerisation. We have so far developed a model which describes the three-dimensional spatial variation of the ink polymerisation via incorporation of the polymerisation kinetics, printing strategy and UV attenuation throughout the process. We have demonstrated how this model can be used to predict the distribution of ink residuals, through which optimum printing strategies can be designed and assessed to balance the need for high product quality and minimised costs in inkjet 3D printing. The next step is to incorporate CFD modelling into this model to enable us to obtain a more detailed prediction and optimisation of the inkjet printing process.

Development and study of a model of the 2PP process

One of the difficulties with the 2PP technique is the relatively narrow range of acceptable process parameters where solidification can take place, without damage to the resulting structure. Based on experimental work in this Programme, we previously discovered a remarkably simple relationship for the damage threshold, which, however, is still not fully understood. Therefore, over the past year, starting with an existing model for a single voxel we have developed a new model that can produce results for any pattern of light exposure in space and time. Going forward, the plan is to relate the output of the model to experimentally measured quantities for validation, and to develop approximations that may allow us to avoid costly computations and achieve a better understanding of 2PP.

Research challenge 3 (RC3)

Controlling connectivity and anisotropy for enhanced functionality

***Aim:** To be able to control the connectivity of additively manufactured objects to enable full functionalisation*

This research challenge aspires to achieving functionality in 3D, where (informed by RC1/RC2) the rules for macroscopic inter and intra layer deposition of functional materials is studied. We aim to establish an understanding of the temporal and spatial manner in which materials are deposited and consolidated to create homogenous and isotropic structures that construct into the final product. By examining the relationship between ink formulation, product performance and processing strategy, it is possible to uncover principle drivers for creating high quality products. The main thrusts have been:

- Key steps have been taken in developing materials, specifically for AM processes, to optimise the interface adhesion between layers. We have adopted the approach of utilising pre-synthesised hyperbranched materials to incorporate the main functionality of key chemistries that can deliver high levels of ink conversion and better interlayer bonding with minimised viscosity increment. The success of this will improve the connectivity and anisotropy from the chemistry aspect.
- For material jetting, we aim to uncover the principal drivers in achieving isotropic products. We have conducted a systematic assessment of how inks polymerise and behave during the layer-wise manufacturing process. By collaborating with RC1 and RC2, we were able to establish the correlation between chemical kinetics and processing parameters. This enables us to optimise the connectivity and anisotropy from the manufacturing aspect.
- Understanding of 2PP is challenged by the quantum nature of the process, with the requirement for absorption being a combination of quantum cross section, intensity of incident photons, density of states and conservation rules. We have taken the first steps to understand the relationship between printing strategy (density of the 'hatch', repetition of exposure, etc.), diffusion (both of radicals and oxygen), material reactivity and photoinitiator efficiency. This understanding has been captured in a simple model that replicates the main features of our observations.
- With the support of high resolution chemical profiling techniques from RC1, we were able to understand the mechanics of combining different materials during multimaterial printing. Whilst relatively simple for single materials, with multimaterial AM, it is important to understand whether different materials form reliable bonding at the interfaces or if different materials excessively interpenetrate. Our current findings indicate that different materials do indeed interpenetrate (interphase), but further work is required to understand the degree of bonding and whether copolymerisation occurs.
- In year three, we expanded the multimaterial AM processes by introducing projection micro sterolithography (PμSLA), which has enabled us to manufacture devices at the scale of a few hundred microns. This fills the manufacturing gap where devices are too big to be printed by 2PP, but too small to be printed by material jetting.

The next stage for RC3 will focus on the control over multimaterial deposition, which will be supported by progress in both chemistry and manufacturing aspects. We will explore how the predictive algorithms developed in RC2 can be used to generate control over the material microstructure and guide when and where we should place different materials. In particular, we will use our deepened control/understanding to avoid flaws such as sagging or microflows that tend to appear when the deposition process is suboptimal. This will be particularly important during multimaterial, multi-layer co-deposition (RC4) and will be required to control dissimilar materials, particularly when these materials are not miscible or prefer not to bond with each other. We will begin to consolidate this control to drive the manufacture of devices that are able to sense or deliver.

Auditory ossicles reconstructed using micro CT data and PμSLA

3D macromolecules

The research in RC3 centres around attempting to improve the connectivity between printed droplets and layers, and hence reduce anisotropy in printed articles. The addition of pre-polymerised materials, in the form of hyperbranched (HB) polymers, into ink formulations for inkjet and 2PP has provided some interesting results. In the case of 2PP, processing parameters were found to be less limited than with conventional inks. Additionally, printed articles were found to have reached higher conversion to polymer resulting in a more consolidated structure with less need to remove unreacted starting material. A journal article is in preparation detailing the effect of HB polymers in an inkjet formulation. The library of available HB materials has been expanded and a journal article describing the improved synthesis of these materials is being prepared. The next stages will focus on the use of HB materials in a number of different formulations, exploiting their thermal properties to find suitable applications.

Two photon polymerisation (2PP)

2PP is a promising route for the fabrication of structures with both high design freedom and sub-micron resolution. There are, however, a number of issues preventing its use for production, including a lack of understanding of process conditions, a lack of predictable isotropic property development and a limited capacity for multimaterial manufacturing. Over the last year we have begun to tackle these problems and importantly we have developed a better understanding of the process parameters. This has allowed 2PP to print multimaterial structures capable of retaining their shape in the long term, showing that functionalisation of these structures can be achieved both by *in situ* and post deposition strategies. Follow on studies are now ongoing to investigate the electrical and optical properties of the structures that have been produced via these methodologies. A systematic study of the effect of printing parameters on the properties of the printed structures has now been completed and this data is informing modelling of the 2PP process. These predictive models will ultimately lead to greater understanding and control, allowing us to exploit this technique to its fullness.

Multimaterial jetting and projection micro stereolithography (PμSLA)

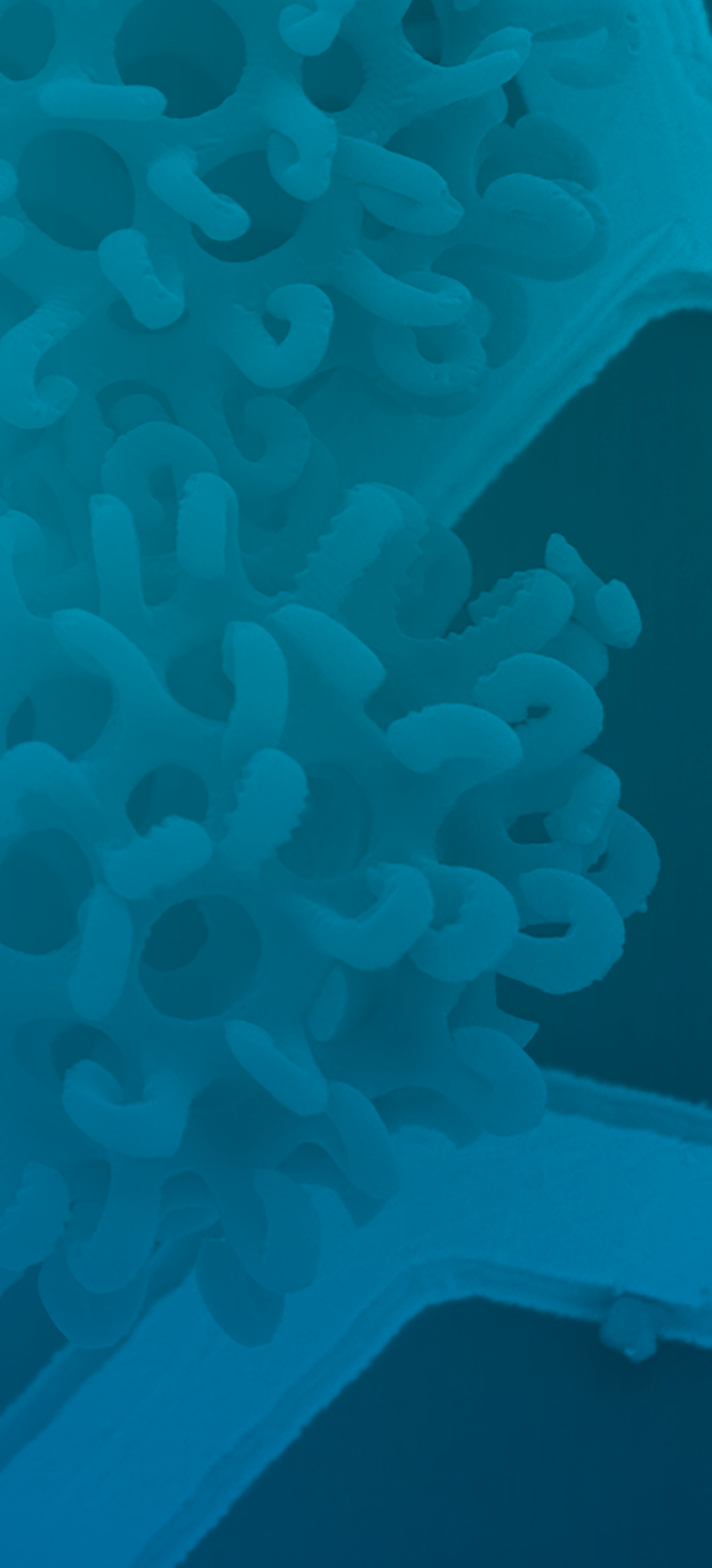
In year three, we introduced PμSLA, a new type of high-resolution AM technique. We are currently building a bespoke multimaterial printing unit to adapt a commercially procured PμSLA system from Boston Micro Fabrication Ltd., which is expected to be running in early 2021. This year we also successfully expanded our functional material database for both multimaterial jetting and PμSLA processes, which has allowed us to manufacture high resolution devices for biomedical and metamaterial applications. In the coming year, we will complete the multimaterial printing unit for PμSLA and will explore a new formulation direction: colloid based ink formulation, to enable the material jetting of high molecular weight molecules, which can significantly improve the final product's performance such as heat resistance, elasticity and mechanical strength. We are also planning to finish building a new multimaterial jetting system that is capable of printing high viscous ink formulations, which will allow us to explore and develop high performance formulations for the material jetting process.

Functional inkjet materials

Materials with advanced functionalities are necessary for the inkjet fabrication of novel devices. Transparency, electrical conductivity, and soft mechanical elasticity are all functionalities of interest for new inkjet materials. This project has investigated these in combination, seeking new understanding of the relationships between formulation, printability, processing, and final properties. PEDOT:PSS is a conductive polymer known for high transparency and conductivity, and is used in optoelectronic devices as a conductor or semiconductor element. Improvement of PEDOT:PSS for inkjet is an active field of study and this project leverages the High Throughput Screening methods developed by CfAM to rapidly investigate novel formulations of PEDOT:PSS inks. We are currently investigating formulations that induce UV-driven conductivity improvements during printing. This project is also leading collaborations, with partners internal and external to the Programme, to develop new concepts for inkjet printing of soft (<10 Kpa) or elastic (>1200 % strain) materials, designing and characterising inks composed of 1) low temperature degrading crosslinkers, and 2) multiphase elastomer inks.

Synthesising elastomeric materials for inkjet printing

There is an increasing demand for next generation AM materials which exhibit elasticity, self-healing capability and mechanical robustness. We aim to prepare a colloid ink from polyurethane-polyethyleneimine (PU-PEI). The colloid nature will allow an increased content of high molecular weight (HMW) polymer in the ink without significantly increasing ink viscosity, allowing it to be inkjet compatible. HMW linear polymer networks exhibit high elastic elongation, and thus this colloid ink will allow inkjet printing of materials with higher elasticity than is possible when using a single-phase ink. A polyurethane will be synthesised from its components to yield a colloidal suspension, as per the literature. An aqueous solution of branched PEI will be blended with the newly synthesised PU emulsion, stabilised and prevented from premature cross-linking using ammonia hydroxide. This results in a colloidal complex which can be easily inkjet printed as is, or further reformulated to customise the printed properties. Later, we aim to vary the colloidal complex chemistry to yield further printable elastomers exhibiting additional functional properties.



2PP structures printed on glass and then transferred onto a copper grid

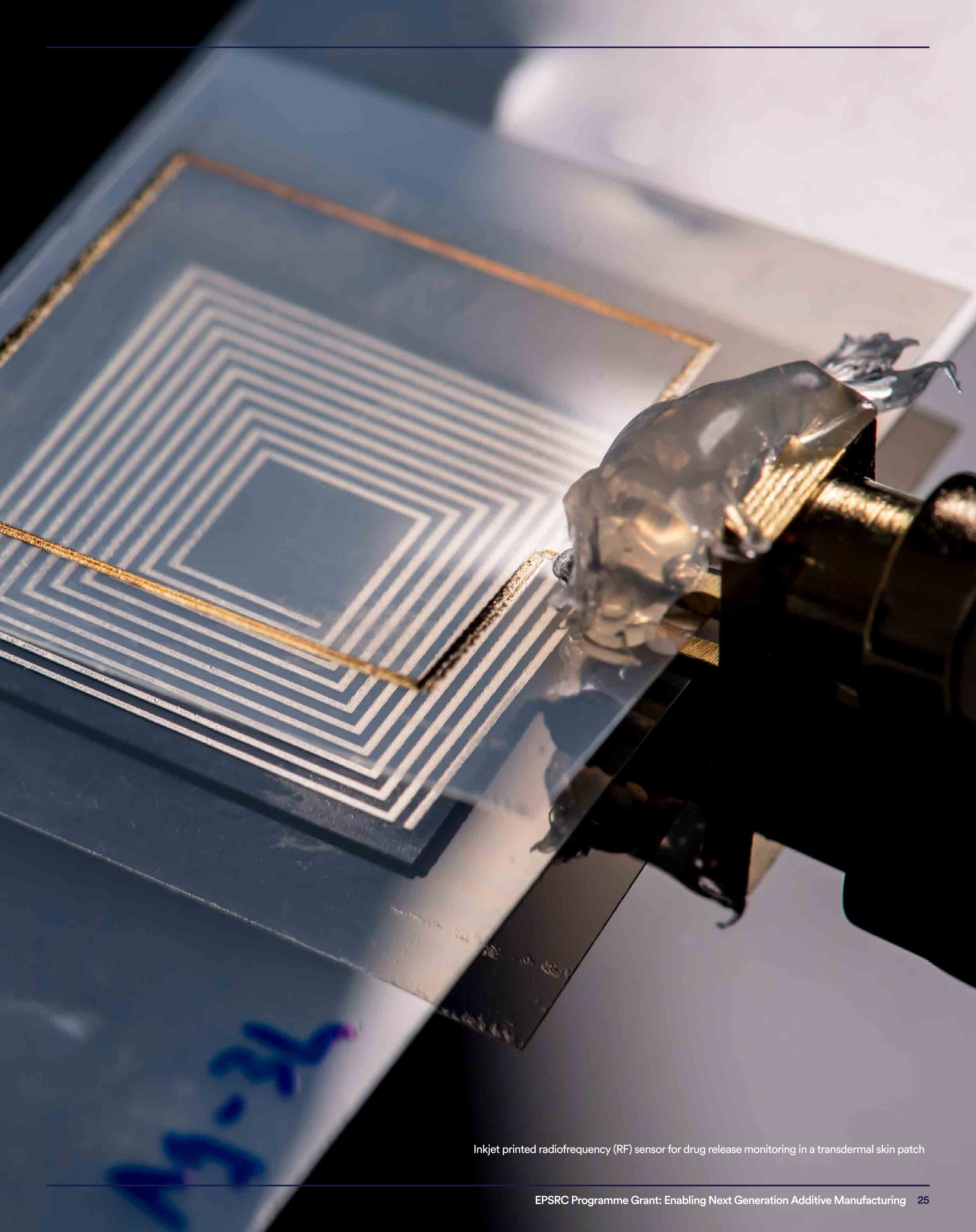
Research challenge 4 (RC4)

Controlled co-deposition of multimaterials

***Aim:** To investigate and demonstrate strategies for the macroscale co-deposition of functional and structural materials via piezo driven jetting, high temperature metal jetting and functionalised multi-photon techniques*

Looking at the controlled co-deposition of multimaterials within electronic and healthcare exemplars, RC4 is the culmination of many of the activities within the Programme. RC4 provides macroscale demonstrators of multi-deposition techniques and aids in the production of samples for validating models and characterisation. We began with a focus on functional material development and their processing requirements. This required modification of surface chemistry and material constituents for successful processing by multiphoton, inkjet printing, PμSLA, volumetric additive manufacturing and metal jetting. These materials and processing methods are now being implemented across a range of demonstrators including (but not exclusively):

- Working with colleagues in Physics, we have established a complete strategy for inkjet printing and post-deposition functionalisation of low dimensional materials for the fabrication of electronic demonstrators. We have successfully printed and characterised a series of 2D materials including Graphene and Hexagonal Boron Nitride (hBNs) and are now working with an extended material set including photo-active materials, such as Indium Selenide (InSe), and PEDOT:PSS for the potential production of light emitting devices and photo-sensing.
- We have developed a conductive gold ink formulation with structural integrity for printed electrodes in 2D & 3D, which will enable the construction of interconnects between 3D stacked functional electronics for miniaturised devices with high density of electronic components. In addition, piezoelectric ink formulations are under development for energy harvesting devices.
- Multimaterial healthcare devices, where we are investigating the fabrication of pre-vascularised bioactive implants for the regeneration of tissues, targeting bone tissue in the first instance. We are multimaterial jetting biodegradable polymers that can support cell growth, and sacrificial inks that aid the fabrication of complex shapes alongside bioactive inks (growth factors) that provide signals for the regeneration of bone. So far, the printing parameters of the structural, sacrificial and bioactive inks have been optimised and materials have been successfully co-printed alongside scaffolds with microchannels that mimic vascular arterioles.



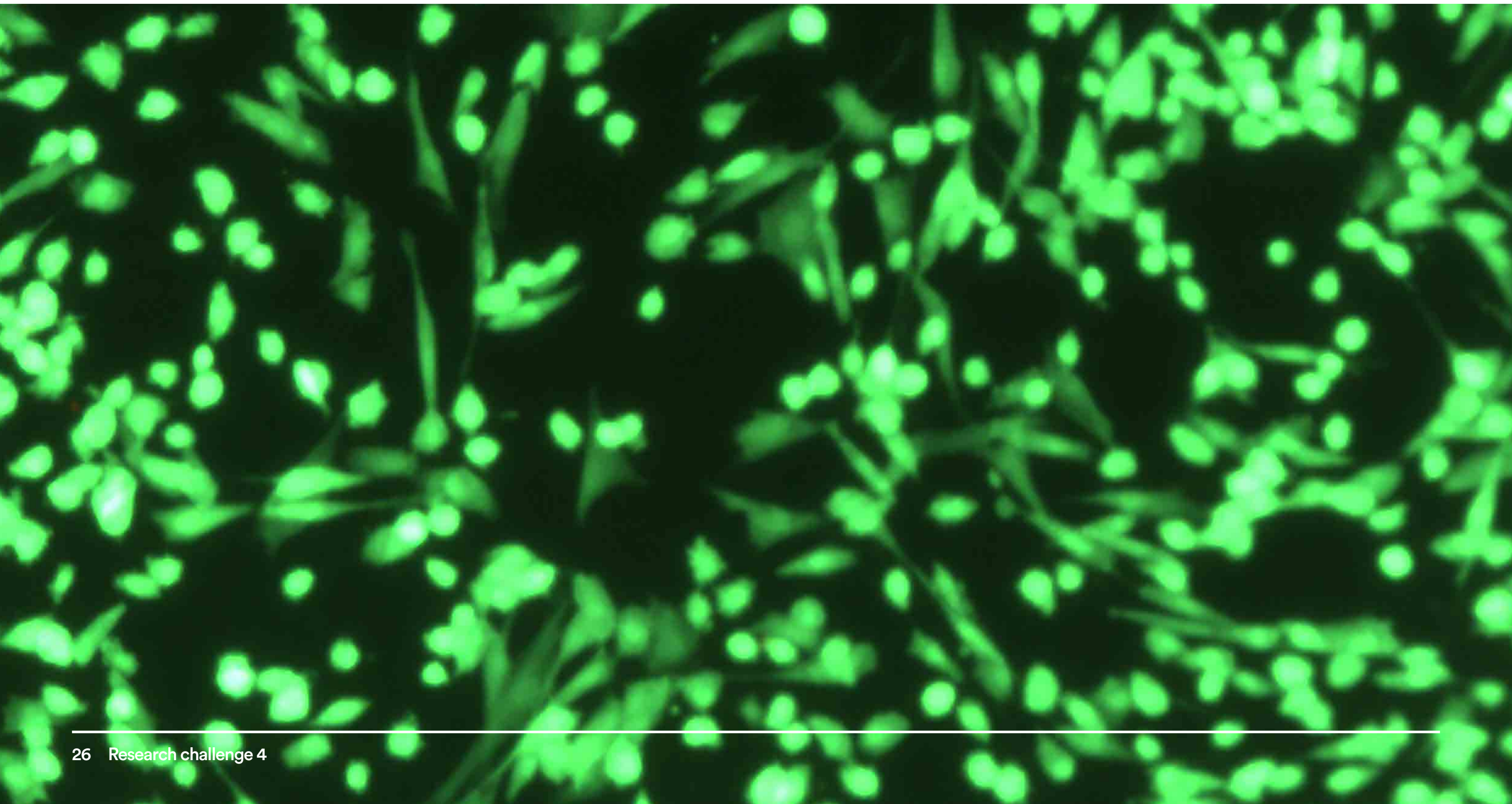
Inkjet printed radiofrequency (RF) sensor for drug release monitoring in a transdermal skin patch

Functionalisation of low dimensional materials for electronic devises

Further experimental and theoretical studies have been carried out on inkjet printed graphene. Detailed electrical and structural characterisation was undertaken using transport modelling, including inter flake quantum tunnelling transport and percolation dynamics. This study revealed that the electrical properties are strongly influenced by the flake packing fraction and by complex meandering electron trajectories, which traverse several printed layers. Apart from the graphene/hexagonal boron nitride field effect transistor, we also demonstrate that inkjet printed graphene can be used to replace single layer graphene to provide ohmic contacts to 2D metal chalcogenides, such as an InSe phototransistor. Further work is also underway to develop a strategy to control the morphological properties of multimaterial interfaces, which in turn affects charge transfer and will enhance device performance for sensing and optoelectronic applications.

Conductive gold ink formulation for electronic demonstrators

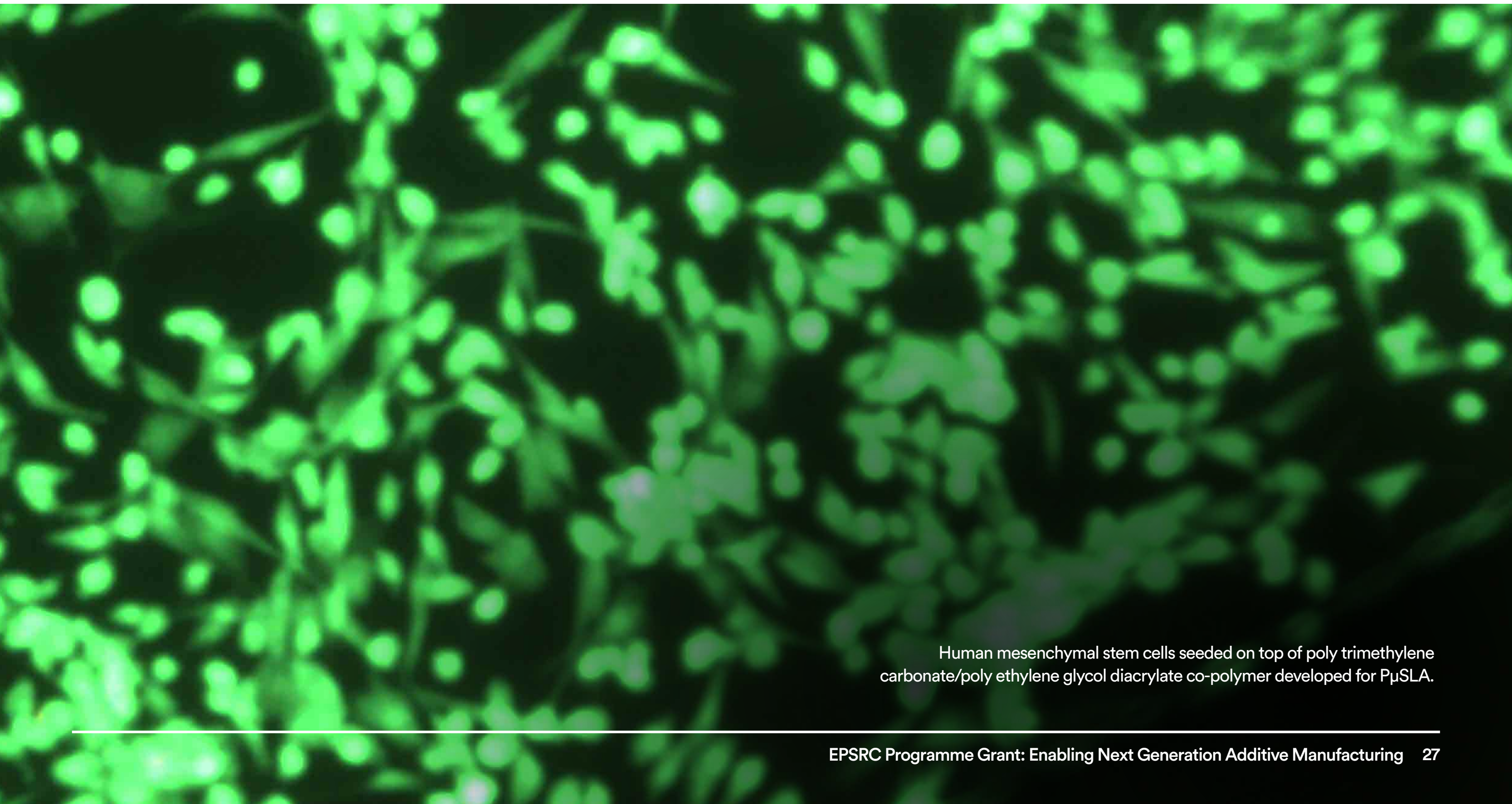
Additive manufacturing accelerates the development of 3D printed multifunctional electronics by enabling rapid prototyping, design freedom, and high-density assemblies of electronic components. We have successfully developed a thiol-based gold nanoparticle-based conductive ink formulation that provides high conductivity, structural integrity of printed layers using a cohesion enhancer, a post-processing method compatible with flexible substrates, and durability in response to bending for flexible printed electronics application. As an electronic demonstrator, we designed a radiofrequency (RF) sensor that can monitor drug release in a transdermal skin patch. The feasibility of the inkjet printed RF sensor was demonstrated by monitoring its resonant frequency change as a function of different drug loading in the inkjet printed drug-containing layer. We will continue to develop electronic materials and provide further electronics demonstrators including an RF drug release monitoring sensor for a transdermal skin patch, a Surface Enhanced Raman Scattering (SERS) sensor prepared via 2PP (RC3&4), and piezoelectric energy harvesters.



Bio-fabrication of vascularised bone

Osteoporosis is a condition resulting in an increased risk of skeletal fractures due to a reduction in the density of bone tissue. Fragility hip fractures are the most serious in terms of cost and morbidity and therefore finding therapies to regenerate bone after a fracture has been the focus of Regenerative Medicine therapies for several years. However, one of the main challenges is being able to mimic the microstructure and vasculature of bone in order to fabricate functional tissue.

The aim of this project is to manufacture pre-vascularised functional bone mimics that can help in the regeneration of bone tissue by providing an adequate environment (microstructure) for the cells to regenerate the tissue. To achieve this, we have synthesised a biodegradable amphiphilic polymer that mimics the cell's extra cellular matrix due to its capacity to retain high volumes of water after crosslinking. We also developed a bio-ink that is composed of this polymer and a gelatine-based material to enhance cell adhesion and proliferation. In collaboration with our partners at the Lawrence Livermore National Laboratory (LLNL) and RC3, we are currently working on optimising the parameters for printing osteons (bone functional units) with this ink using PμSLA. The next steps are to print the osteons and carry out *in vitro* cell culture testing to demonstrate the functionality of the printed osteons.



Human mesenchymal stem cells seeded on top of poly trimethylene carbonate/poly ethylene glycol diacrylate co-polymer developed for PμSLA.

Aligned doctoral research

Developing the next generation of researchers and leaders of AM is a vital part of the Programme's work. Our high calibre doctoral research students work on a variety of projects ranging from the electronic properties of graphene to controlling metal jetting processes and developing responsive materials for controlled drug release.

Negar Gilani
(started October 2018)

Control of high temperature drop-on-demand metal jetting through numerical modelling and experiments

Metal jetting is the latest generation AM technology which is opening up new opportunities for producing intricate metallic components. The drop-on-demand approach has the capacity to produce molten microdroplets (<70 μm) at high temperature (up to 2000 °C) to form single and multimaterial objects. This project aims to understand the 3D deposition of high temperature metallic droplets through an integrated computational experimental approach. Results include:

- a Finite Element (FE) model has been established to simulate solidification and cooling of single and multiple droplets during droplet deposition, which was validated through experiments
- using the FE model, various features observed in the MetalJet experiments were explained, including the reasons for a lack of adhesion at the interface, etc
- through virtual experiments, solutions to rectify the problems were evaluated, which provided the basis of a more targeted experimental investigation
- these solutions were applied to Sn and Ag, and encouraging outcomes were obtained

The future focus is to 1) publish these results, 2) combine the established FE model with a Cellular Automata model to simulate the grain nucleation and growth during solidification, 3) to validate the model with experimental observations, and 4) expand the range of materials to be deposited.

Jonathan Gosling
(started October 2018)

Electronic properties of graphene-based electronics

Inkjet printing of 2D materials involves depositing individual flakes into a matrix form. Many features of printed heterostructures influence the performance of conductive components, from the disorder of flakes to the properties of surrounding dissimilar materials. The electrical properties of dissimilar materials were predicted by quantum transport simulations, including electronic scattering by charged impurities, defects and phonons, and were used to understand experimental measurements.

We developed (and published) a model that explains charge transport in graphene-based heterostructures, including percolation and disorder effects, which accurately reproduces the observed properties of ink jet printed graphene. The size and density of flakes, and amount of disorder were found to have significant effects.

This model will now be expanded to include the transport of charges with different polarity (JSPS Fellowship 2019-2020) and the properties of optoelectronic graphene based devices, to inform the next stages of 2D material printing.

Eric Lehder
(started October 2018)

Optimising the geometry of a fracture healing assembly that includes a cell seeded scaffold and a stiffness graded auxetic fixation plate

Additively manufactured scaffolds with biocompatible, biodegradable and bone matched mechanical properties could improve the outcomes of bone healing and regeneration. To keep the bone-scaffold assembly stable while osteo-blast cells develop into mature bone, a fracture fixation plate is necessary. Two promising solutions for the problem of stress shielding with stiff plates are stiffness grading and auxetic structures.

This work aims to develop a computational method to optimise a triply periodic minimal surface (TPMS) geometry bone regeneration scaffold, and a stiffness graded auxetic fixation plate. The former has the objective of maximising the regenerative capacity, while the optimal plate minimises the stress shielding. A curvature dependent model was used to optimise the scaffold geometry, while a mechanical performance finite element model was used to optimise the plate geometry.

In the upcoming year, cell culture experiments will be undertaken to complement the results obtained with the cell growth model. Moreover, a compression strain test will be done to verify the plate optimisation.

Maria Inês Evangelista Barreiros
(EPSRC Centre for Doctoral Training in Additive Manufacturing, started February 2019)

Design and manufacture of drug delivery platforms

Pharmaceutical formulation development is a very lengthy and difficult process. This is especially difficult when dealing with poorly soluble drugs, which is the case for most new chemical entities (NCEs). Research in 3D printing of oral solid dosage forms has shown that it is possible to manufacture tablets with less excipients, higher drug loadings and tuneable release profiles. While one of the drawbacks of 3D printing is the smaller number of materials that can be used with it, it also gives the opportunity to explore non-pharmaceutical grade materials that are actually biocompatible and that haven't been explored for drug delivery.

Therefore, this work proposes the development of simple formulations for the release of different drugs in oral solid dosage forms, while controlling the drug release profiles based on different polymer carriers and change in geometry, and comparing the printed tablets manufactured by two different printing techniques – material extrusion and material jetting. It also looks at the development of drug delivery implantable devices by exploring the use of new materials for 3D printed pharmaceutical applications.

In the year ahead, the plan is to produce and characterise formulations to be used in both material extrusion and jetting, and to optimise the printing parameters. *In vitro* dissolution studies are to be performed as well. Following on from my published work on the characterisation of printed implants and *in vitro* drug release studies, I will consider the feasibility of using similar polyurethanes with different mechanical properties as drug carriers on implantable devices.

Eva Kingwood
(Started October 2017)

3D inkjet printing of multimaterial pulsatile drug delivery implants

3D printing has shown potential in the field of drug delivery due to its high geometrical freedom and ability to print medical devices with personalised drug combinations and doses. This project aims to exploit these advantages to material jet two biocompatible and bioerodible inks within the same implant, in order to exhibit a pulsatile release of the drug. UV-crosslinkable poly(trimethylene carbonate acrylate) and poly(carbonate)-based inks have been developed and printed into three novel implant designs, with OrbiSIMS analysis confirming clean surface interactions between the two cross-linked materials and no bleeding of the two materials. The upcoming aims of the project are to conduct *in vitro* drug release studies of each implant design to determine which designs offer clean and sharp pulses of the fluorescent dye Rhodamine-b.

Kevin Bandeira
(EPSRC Centre for Doctoral Training in Additive Manufacturing, started February 2019)

3D printable implants for long term controlled drug release

The aim of the project is to develop a controlled drug delivery platform utilising a printable combination of a photo-reactive monomer and an active pharmaceutical ingredient (API). Employing 3D inkjet printing and UV irradiation, we were able to successfully print 3D structures loaded with ibuprofen. Dissolution studies are in progress and the dosage forms require characterisation over a 24-hour period to validate the release of ibuprofen. Within the coming year, investigation of potentially implantable API-delivery platforms will be explored for the purposes of providing a novel manufacturing route to implantable therapeutics. The system studied will be relevant for the purposes of treating patients with General Anxiety Disorder (GAD).

Kristian Plender
(started October 2019)

Novel approaches to the long term release of biomacromolecules

The long term delivery of therapeutic biomacromolecules is highly desirable, however retaining activity until elution from a delivery device is a multi-factorial challenge. The main project aim is to develop formulations with relatively high biomacromolecule loading that can be 3D printed to produce an implant for sustained release over extended periods of one to three or more months. 3D printing allows a high degree of flexibility regarding implant geometry and control over the spatial location of bioactives, to optimise the subsequent release profile.

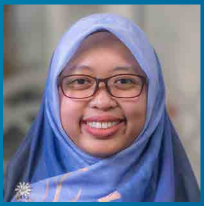
Current experimental work has been focused on establishing the activity retention of alkaline phosphatase (ALP; a model protein) within biodegradable polymer formulations, which are printable using an inkjet system. The upcoming year will focus on optimising formulations that can protect ALP during the printing process and subsequently in the implant. The structures fabricated will be used to conduct release studies to determine ALP elution and activity retention.

Joseph Sefton
(started October 2019)

Production and use of oligomers in additive manufacturing

This project focuses on the delivery of novel, reactive polymeric materials for use in photo-initiated inkjet printing (IP). Formulations used in IP must meet strict viscosity parameters and must react rapidly to form a self-supporting structure. We have proposed vinylic oligomers (reactive, low molecular weight polymers) produced via catalytic chain transfer polymerisation as candidates to modulate the viscosity and reactivity of IP formulations.

We are currently investigating the addition of oligomers of alkyl methacrylates to a well characterised diacrylate formulation. High throughput viscosity analysis will allow rapid identification of 'printable' formulations, allowing us to study the impact of oligomer structure and loading on formulation viscosity. The selected formulations will be used in IP and the reactivity and final polymer properties will be examined. We aim to use the findings of the project to assist in the development of biomedical IP formulations by synthesising and formulating specific oligomers, in collaboration with other Programme members.



Nur Rofiqoh Eviana Putri
(started December 2019)

Additive manufacture of vascularised bioactive scaffolds for bone tissue engineering

One of the long standing challenges for the success of bone tissue engineering *in vivo* is the slow vasculature invasion into scaffolds/implants, which leads to cell death in large scaffolds due to the lack of nutrients and oxygen diffusion. To solve this challenge, tissue engineering has developed several methods to fabricate capillaries within the scaffolds before implantation. However, the challenges of replicating the complex architecture and functionality of the capillary bed are immense, and successes are scarce.

This work aims to create bone tissue analogues with integrated vasculature using inkjet 3D printing, biodegradable materials and precise positioning of growth factors. An amphiphilic diacrylated triblock copolymer of poly(trimethylene carbonate) and poly(ethylene glycol) combined with Gelatin-methacryloyl was successfully prepared. The ink mixture was shown to be homogeneous and water soluble. In the next steps, exploration of the vascularised scaffold fabrication and modification of the copolymer will be conducted to conjugate the bioactive components into the materials.

Tien Thuy Quach
(started December 2019)

Novel micro/nano scale characterisation of interfaces in multimaterial additive manufacturing

A primary concern in terms of enabling the next generation of AM systems is the difficulty of mixing and interactions at the interfaces of functional and structural materials, due to differences in their physical and chemical properties. To overcome these problems, one of the requirements is the development of interface analysis at the micro/nanoscale. The aim of this project is to advance the fundamental understanding of interface phenomena in multimaterial AM. This will be achieved mainly by developing tailored methodologies related to electron microscopy and various spectroscopies. This project has examined 3D printed multimaterials including a 3D printed ink (gold conductive ink) and an inductor (DragonFly product) by using different techniques such as scanning electron microscopy (SEM) and transmission electron microscopy (TEM), and using ultramicrotomy to cross section and reveal buried interfaces for analysis. In the coming year, this project will continue to develop interface analysis methodologies by using a variety of techniques to investigate 3D printed electronics and pharmaceuticals.

Jonathan Austin
(started October 2020)

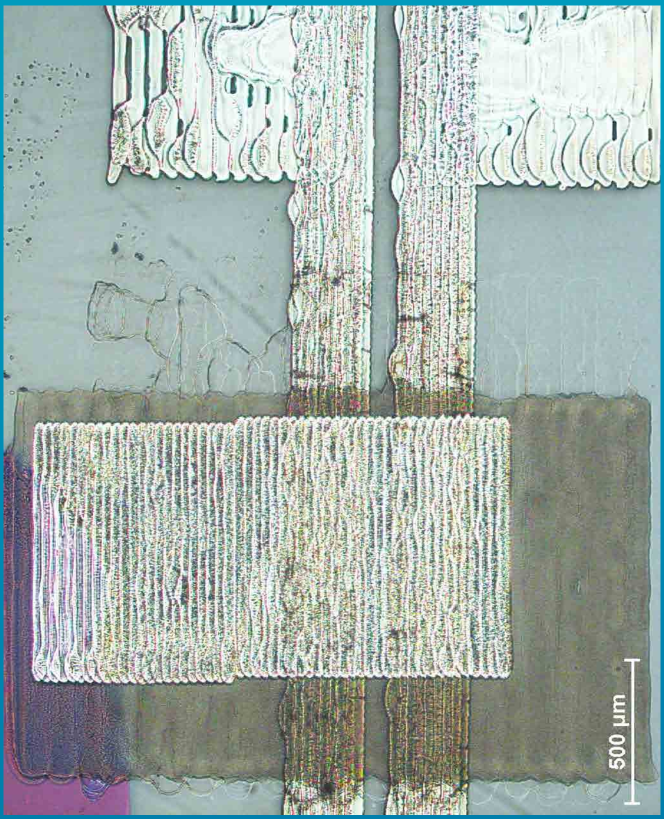
Inkjet printing 0D- and 2D-materials for optoelectronic devices

Devices that can combine the tuneable optical properties of 0D materials (quantum dots and perovskites) with the unique electrical properties of 2D layers (graphene) have recently been considered as promising candidates for the next generation of optoelectronic devices. However, upscaling the processing and manufacturing of these devices remains challenging.

This project focuses on developing inkjet printing 0D and 2D materials and their integration into heterostructures for the production of optoelectronic devices. The initial aim is to manufacture a fully inkjet printed LED comprising an optically active layer of perovskite nanocrystals and a graphene electrode. This device will also include several other materials developed in the Programme such as PEDOT:PSS and gold nanoparticles. The knowledge generated in this study will be used for developing the up-scalable production of devices containing low dimensional materials, such as photon sensors with extended detection range and high photosensitivity. Inkjet printing low-D materials will enable the development of flexible/transparent optoelectronics.

Focus on

Despite the Covid-19 pandemic and the loss of several months of laboratory work, there has been a tremendous amount of progress and we'd like to take this opportunity to focus on three particular developments within this year's Annual Review:



2D materials

The Programme's work on the additive manufacture of 2D materials has produced some exciting results this year. 2D materials have unique structural and electronic properties with the potential for transformative device applications. Additive manufacturing with inks containing 2D material flakes is a promising solution for producing high quality large area hetero-structures. We have been able to show the deposition of electrically conductive graphene layers and insulating hexagonal boron nitride layers for devices such as field effect transistors and photon sensors. We also demonstrated, for the first time, that inkjet printed graphene can successfully replace single layer graphene as a contact material for 2D metal chalcogenides to form Ohmic contacts. The experimental results obtained are supported by theoretical models of flake-to-flake charge transport. The properties of interfaces in 2D based hetero-structures require particular attention and the sequential deposition of layers results in the intermixing of materials, as was revealed by ToF-SIMs [Adv Func Mater 2020]. Pristine, encapsulated and quantum dot functionalised graphene sheets can have differing electrical properties, due to the differing nature of the surrounding materials. We found that there was a universal connection between the carrier mobility and the variation of electrical conductivity with carrier density. Phenomenological relations provide a way to predict *a priori* all key transport parameters of graphene devices. First principles-based simulations, which considered the altered electrostatic environment induced by surrounding materials, were used to verify the resulting relations, and understand their origin [Comm Phys 2020].

We aim to use these insights into the behaviour of 2D materials to develop other electronic and optoelectronic applications such as digital processors with multiple vertically stacked 2D material layers.

Optical microscopy image of a field effect transistor containing an inkjet printed graphene channel

Reactive prodrug ink formulations

3D printed personalised medication can result in improved compliance and patient treatment outcomes, but bottlenecks to their development include finding formulations that provide a choice of drug loading and release rate, that are tuneable, and avoid the need for surgical removal. In this project we used 3D inkjet printing to explore manufacturing drug delivery freedoms [Adv Ther 2020]. A reactive prodrug was used which can polymerise into drug-attached macromolecules during 3D printing and by tuning the hydrophilicity, hydrolysis can be facilitated or hindered, which in turn controls drug release. To demonstrate this approach, ibuprofen was attached to 2-hydroxyethyl acrylate through a cleavable ester bond, formulated for inkjet 3D printing, and then printed to produce a solid dosage form. This allows a much higher loading than is usually achievable, in this case up to 58 wt%. Of equal importance, the 3D inkjet printing freedoms mean that the drug delivery device was highly tuneable: by selecting spacer monomers to adjust the hydrophilicity; through geometry and by spatially varying the components. Consequently, hierarchical release systems were created bespoke, from the molecular to macro. This approach represents a new paradigm for the formulation of printable inks for drug-loaded medical devices.

New Co-I joins the Programme

Dr Lyudmila Turyanska (Lyuda) formally joined the Programme as a Co-I in early 2020 and has already had a big impact on our work, across the RCs. Her research is focused on the study of functional low-dimensional materials, which crosses the boundaries between Physics, Chemistry and Pharmacy. She has successfully developed research projects on the growth and fundamental properties of 0D and 2D materials; hybrid low dimensional composites for optoelectronic devices; and multifunctional nanoprobes for medical imaging and nanomedicine. She has an MSc in Chemistry and PhD in Physics, which has enabled her to successfully develop interdisciplinary projects. Lyuda has been the recipient of several prestigious awards and fellowships including an Anne McLaren Fellowship, MRC Discipline Hopping Fellowship and a Commendation Award at the House of Commons Britain's Early-Stage Researchers reception.

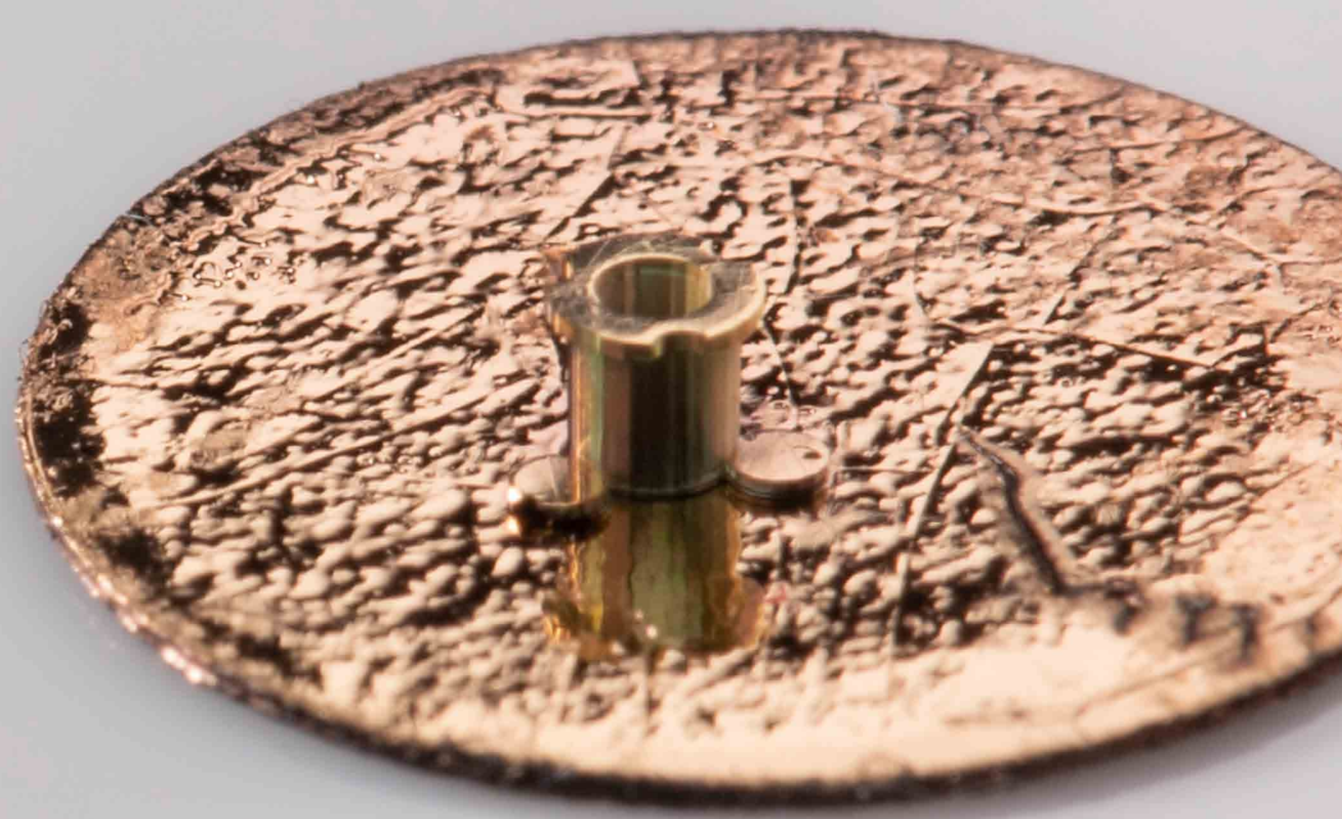
Since joining the Programme and the CfAM at Nottingham, her research has focused on the development of novel nanomaterials, including the synthesis of perovskite nanocrystals and their exploitation in devices [Adv Func Mater, 2020 a], fundamental studies of graphene and graphene-based heterostructures [Adv Func Mater, 2020 b; Commun Phys 2020], and applications of nanoscale materials for drug delivery [ACS Appl Mater Interf, 2020]. She has been contributing to the work of RC4, mostly providing expertise on material characterisation and their integration into electronic devices. She also collaborates with colleagues in RC2 and RC3. Her current research focus is on the integration of nanomaterials within additive manufacturing processes, which could offer opportunities for *in situ* and post-deposition control of material properties, as well as design freedom. This could provide a route to the up-scalable fabrication of multimaterial structures with properties tailored for specific applications.

Outputs

This year, due to Covid-19 related restrictions, we have not been able to carry out our normal programme of research visits, outreach activities and summer studentships. However, we have been able to continue publishing papers in high quality journals, apply for funding and present at conferences and seminars.

Publications

The Programme has a strategy of publishing high quality papers in leading journals, and has applied strict standards to our experimental results and manuscripts, which significantly increases the development time. However, this strategy is starting to bear fruit and we are pleased to present 18 published papers aligned with the theme of the Programme, a further five submitted for publication, alongside 38 in preparation.



Grommet (ventilation tube) used for the treatment of middle ear infections in children. Printed using PµSLA and gold plated for characterisation by SEM

Published

Design of highly stabilized nanocomposite inks based on biodegradable polymer-matrix and gold nanoparticles for inkjet printing. Belen Begines, Ana Alcudia, Raul Aguilera-Velazquez, Guillermo Martinez, Yinfeng He, Gustavo F Trindade, Ricky Wildman, Maria-Jesus Sayagues, Aila Jimenez-Ruiz & Rafael Prado-Gotor. Scientific Reports. November 2019. doi.org/10.1038/s41598-019-52314-2

Delivery of Temozolomide and N3-Propargyl Analog to Brain Tumors Using an Apoferritin Nanocage. Kaouthar Bouzinab, Helen S Summers, Malcolm F G Stevens, Christopher J Moody, Neil R Thomas, Pavel Gershkovich, Nicola Weston, Marianne B Ashford, Tracey D Bradshaw, Lyudmila Turyanska. ACS Applied Materials & Interfaces. February 2020. doi/10.1021/acsami.0c01514

Bouncing off the Walls: The Influence of Gas-Kinetic and van der Waals Effects in Drop Impact. Mykyta V Chubynsky, Kirill I Belousov, Duncan A Lockerby, James E Sprittles. Physical Review Letters. February 2020. doi.org/10.1103/PhysRevLett.124.084501

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Dr Adja Touré using the BMF nanoArch S130 PμSLA system

Conferences and seminar presentations in year three

Title	Presenter	Event	Date	Venue
Thermal fluctuations in free surface nanoflows	James Sprittles	IAM Seminar, University of Oxford	January 2020	Oxford, UK
On the interaction and forms of adhesion between various substrate materials and molten metal droplets produced by the drop-on-demand technology MetalJet	Nesma Aboulkhair	TMS2020	February 2020	San Diego, California, US
Thermal fluctuations in free surface nanoflows	James Sprittles	DAMTP, University of Cambridge	March 2020	Cambridge, UK
Enabling Next Generation Additive Manufacturing: the 3D deposition of functional materials for the additive manufacturing of smart devices. (keynote speaker)	Richard Hague	International Conference on Research Advances in Additive Manufacturing (RAAM)	March 2020, postponed	Nanjing, China
Multivariate analysis of secondary ion mass spectrometry data	Gustavo F Trindade	SIMS Workshops	May 2020	Nottingham, UK, Online
3D Printing of Medicines: a practical reality for manufacture (keynote speaker)	Clive Roberts	International Conference and Exhibition on Pharmaceutical Sciences and Technology 2020 Bangkok	May 2020	Bangkok, Thailand, Online
The promise of 3D printing in pharmaceuticals and dietary supplements	Clive Roberts	United States Pharmacopeia Conference 2020	May 2020	Online
Enabling Next Generation Additive Manufacturing: research challenge 1	Gustavo F Trindade	Departmental seminar, University of Sao Paulo	July 2020	Sao Paulo, Brazil, Online
Matrix augmentation of mass spectrometry imaging datasets	Gustavo F Trindade	SIMS Workshops	September 2020	Nottingham, UK, Online
Designing and optimising micro/nanoscale characterisation methodologies for next generation multifunctional 3D printed products	Tien Thuy Quach	East Midlands Doctoral Network Conference	September 2020	Leicester, UK, Online
Realising your quantum ambitions - a super position to be in? (discussion panel member)	Mark Fromhold	SPIE Photonex and Vacuum Expo 2020	October 2020	Online

Title	Presenter	Event	Date	Venue
Modelling additive manufacturing by two photon polymerisation	Mykyta Chubynsky	Printing for Fabrication: materials, applications, and processes	October 2020	Online
Processing multiple mass spectrometry depth profiling datasets	Gustavo F Trindade	SIMS Workshops	October 2020	Nottingham, UK, Online
Invited set of seminars on Nanomaterials	Lyudmila Turyanska	University of Lincoln	Autumn 2020	Lincoln, UK Online
Continuum models of nanoscale effects in initial stages of droplet spreading	Mykyta Chubynsky	The annual meeting of the Division of Fluid Dynamics of the American Physical Society (APS DFD 2020)	November 2020	Online
Control of high temperature drop-on-demand metal jetting through numerical modelling and experimentation	Negar Gilani	Material Science and Technology (MS&T20)	November 2020	Online
Bouncing off the walls: the influence of gas-kinetic and van der waals effects in drop impact	James Sprittles	The annual meeting of the Division of Fluid Dynamics of the American Physical Society (APS DFD 2020)	November 2020	Chicago IL, USA, Online
Free surface nanoflows	James Sprittles	Imperial College London	November 2020	Online
Free surface nanoflows	James Sprittles	Lawrence Berkeley National Laboratory	November 2020	Berkeley, CA, USA, Online
Micro/nanoscale characterisation for next generation 3D printed multimaterials (poster)	Tien Thuy Quach	Division of Advanced Materials and Health Technologies (AMHT), University of Nottingham	December 2020	Nottingham, UK , Online
Additive manufacture of vascularised biocompatible scaffolds for bone tissue engineering (poster)	Nur Rofiqoh Eviana Putri	RSC Biomaterials Chemistry Group Annual Meeting	January 2021	Nottingham, UK, Online
Developing micro/nanoscale analyses for next generation multifunctional 3D printed products (poster)	Tien Thuy Quach	Allied Health Professional Postgraduate Research Conference 2021, University of Nottingham	January 2021	Nottingham, UK, Online

Lattice structure printed by PµSLA with unit size of 200µm (front), 400µm (middle) and 600µm (back)

Funding

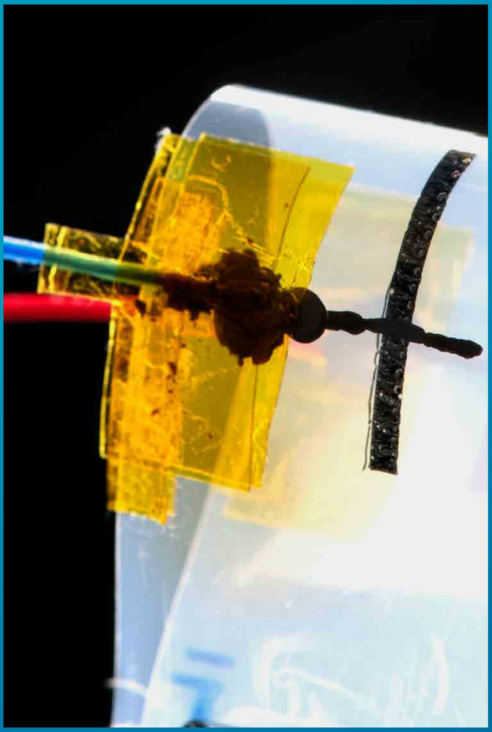
The scale of the Programme has attracted significant additional resource and to date we have leveraged approaching £16m of new funding.

Funder	Title	Person	Value
Geared funding to date			
EPSRC	Prosperity Partnership (BAE) - Intelligent Structures for Low Noise Environments	Tuck/Hague	£715,093 (of £1.7m)
EPSRC	Prosperity Partnership (GSK) - Accelerated Discovery and Development of New Medicines: Prosperity Partnership for a Healthier Nation	Wildman	£562,538 (of £5.5m)
EPSRC	Savi: NSF-EPSRC A Transatlantic Institute for Volumetric Powder Bed Fusion	Tuck/Hague	£254,070
EPSRC	An automated hardness tester and mapper	Simonelli/Aboulkhair	£60,000
EPSRC	Wireless communication with cells towards bioelectronic treatments of the future	Rawson/Hague	£974,695
EPSRC - Impact Accelerator	Scaling up and optimization of the printing process for a patented powder based 3D reactive inkjet technology	He	£62,277
EPSRC - NanoPrime	Design of novel thermal treatments for additive manufactured titanium alloys through high-temperature microscopy	Simonelli	£2,000
EOARD USAF	Complex materials for advanced device fabrication	Wildman/Tuck	£191,170
DSTL	Ink-jet Printing for Fabrication of Tailored Electromagnetic Materials	Tuck/He	£137,078
Industry	Confidential Industry - MetalJet	Simonelli/Aboulkhair	£6,711
Industry	Texas Instruments – three studentships	Tuck/Hague	£243,357
Industry	Confidential Industry - MetalJet	Simonelli/Aboulkhair	£14,605
Industry	Confidential Industry - MetalJet	Simonelli/Aboulkhair	£14,850
Industry	Silicone Jetting Phase 2 Micro-SLA	Tuck/Hague	£356,477
Nottingham UNICAS	Tea for two photon polymerisation	Clark	£12,133
São Paulo RF	3D printing of Micro-Patterned Gaseous Detectors: A route for sensor adaptability and optimisation	Trindade	£20,000
MRC Confidence in Concept (CiC)	Preparing for first in man trials of 3D printed solid dosage forms	Wildman	£76,083
Wellcome Trust	Development of a fatigue detection device to prevent musculoskeletal injuries in football	Ruiz Cantu	£14,900

Funder	Title	Person	Value
Geared Funding to date			
Wellcome Trust	Mechano-chemical integration at the osteochondral interface	Ruiz Cantu	£14,900
EPSRC	Dynamic Wetting & Interfacial Transitions in Three Dimensions	Sprittles	£870,000
EPSRC	Centre for Doctoral Training in Modelling of Heterogeneous Systems	Sprittles	£5,752,474
EPSRC	UK National Quantum Technology Hub in Sensing and Timing	Fromhold	£2,700,000
EPSRC UK Quantum Technology Hub for Sensors and Metrology ATEP, EPSRC DTA	4-year PhD studentship: Computer simulation to optimise the devemopment and deployment of quantum sensors	Fromhold	£100,000
DSTL	CMOS atom chips: a platform for scalable ITAR-free manufacture of fully integrated cold-atom trapping and control systems for quantum sensors	Fromhold	£176,000
DSTL	Design optimisation of low SWaP magnetic field generating, shielding and atom trapping subsystems for quantum sensors of magnetic fields, gravity and rotation	Fromhold	£120,000
EPSRC - Euro MagNET	Magneto-optical studies of all-inorganic perovskite nanocrystals	Turyanska	£50,400
NBCRC	Investigating *in vivo* efficacy of apoferritin-encapsulated antitumour benzothiazole 5F203 against human-derived breast cancer xenografts	Turyanska	£10,000
DSTL	Graphene based UV sensor	Turyanska	£98,000
EPSRC/Industry	iCASE PhD studentship: The Synthesis and Continuous Manufacture of Novel, High Performing Polymeric Lubricants for the Next Generation of Electric Transportation	Irvine	£116,928
EPSRC	Doctoral Training Partnership in Novel Complementary Diagnostics for Improved Therapeutics/Theranostics	Irvine	£356,520
JSPS	Summer Programme 2020	Gosling	£5,800
EPSRC	Equipment grant for a suite of multimaterial AM facilities	Tuck	£1,900,000

Lattice structure printed by PμSLA with unit size of 200μm (front), 400μm (middle) and 600μm (back)

Mid-term review



We were pleased to have the opportunity to show our progress and receive feedback as part of a mid-term review (MTR) by our funder, the EPSRC.

We submitted a report in July 2020 which outlined our vision and ambition, progress to date, added value, leadership and management structures, public engagement activities, and the Programme's plans for the future. Highlighting the scale and activity of the Programme and how this has led to a growing portfolio of impact, the MTR also reported that the Programme had had a successful start, where up to the mid-term we had:

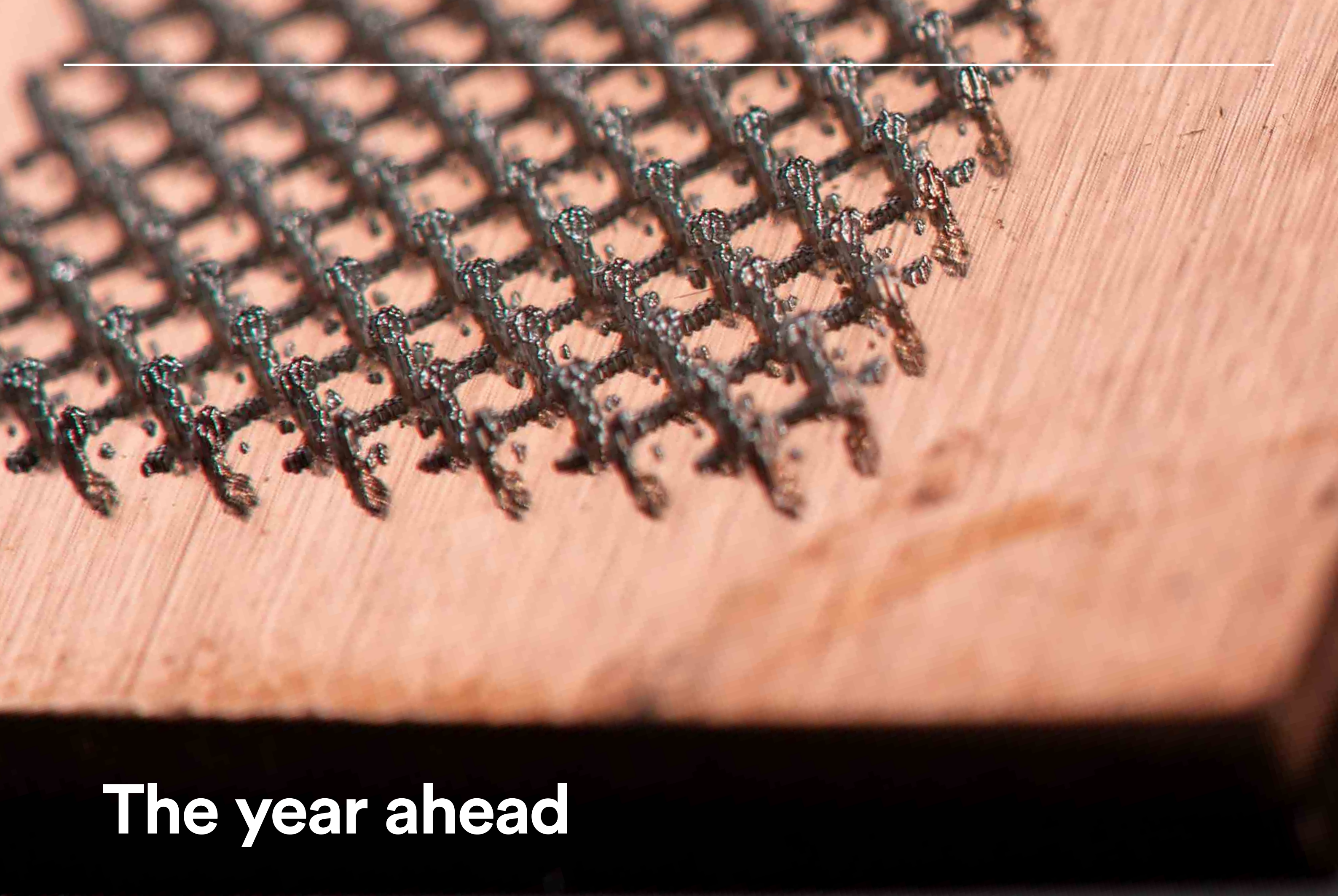
- a full complement of multidisciplinary staff, within and across our institutions (with scholarships from our industry partners and institutions in support of the Programme)
- strong and growing interactions across partner institutions and academic/industrial collaborators
- fully operational RCs across the four main process families

Overwhelmingly, the external peer reviewers and review panel commended what they felt was a very strong team and on the quality of the research, which was noted as having transformative potential. There was particular praise for the added value of the project and how leverage had been utilised. The excellent use of flexibility in resource allocation was noted, and it was observed that the management and leadership was strong at all levels. The panel also felt that four prestigious junior research fellowships was highly commendable. The panel's recommendation to the EPSRC was that the Programme was making strong progress and should continue, giving an overall score of 9 ("Very Strong").

We are obviously delighted to have received such positive feedback, but will continue to challenge ourselves by continuing to produce the highest quality research. The ambitious nature of our Programme also means that there is much work to be done to fulfil the legacy of the Programme and its potential impact. As such, securing future funding to enable us to retain our most talented researchers and continue to lead in this emerging field is clearly of importance. Therefore during the second half of the Programme we will continue to pursue related funding (e.g. EPSRC Centre to Centre call 2020) and strategic applications with EPSRC and others.

Flexible gold electrode printed on polymer

Dr Jisun Im operating the Ultimaker S5 printer



The year ahead

The Programme is running well and to schedule, and we have made exciting advances within the RCs to realise our Programme vision. We have been successfully reviewed by the EPSRC and are confident that we have built up a leading position in the emerging area of multifunctional AM research

As the Programme continues, the role and input of our aligned PhD cohort will also increase (14 from February 2021). These students will be working on projects that align with the Programme's strategy and we will be able to gain significant traction in work that is associated with our application areas of pharmaceutical devices, 3D electronics and (increasingly) regenerative medicine and devices. This development of our PhD students, and of course, post-doctoral researchers, into future leaders and leading scientists/engineers in industry and academia is one of our key priorities.

The ambitious nature of this Programme means that we cannot rest on our laurels and so in year four, we will continue to work across the RCs to develop AM processes such as PμSLA for multimaterial printing, develop a computational framework to maximise the efficiency of printing processes, and use application foci to drive our research. In order to retain our talented researchers and maintain our position as leaders in this field, we will continue to pursue related funding applications with the EPSRC and other strategic partners, and build on the strong relationships with our high profile collaborators. We will continue to communicate our work by publishing in high impact journals, and taking part in outreach and engagement activities, such as presenting at the high profile Royal Society Summer Science Exhibition 2021, which will be a digital event this year.

3D printed lattice of tin on copper using the MetalJet platform

Management structure

Executive Team

Professor Richard Hague
University of Nottingham
Principal Investigator and joint RC1 Lead

Professor Clive Roberts
University of Nottingham
Co-Investigator and joint RC1 Lead

Professor Ian Ashcroft
University of Nottingham
Co-Investigator and joint RC2 Lead

Dr James Sprittles
University of Warwick
Co-Investigator and joint RC2 Lead

Professor Ricky Wildman
University of Nottingham
Co-Investigator and joint RC3 Lead

Professor Derek Irvine
University of Nottingham
Co-Investigator and joint RC3 Lead

Professor Chris Tuck
University of Nottingham
Co-Investigator and joint RC4 Lead

Professor Mark Fromhold
University of Nottingham
Co-Investigator and joint RC4 Lead

Professor Yulii Shikhmurzaev
University of Birmingham
Co-Investigator

Dr Lyudmila Turyanska
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