Diamond Light Source Ltd

2022/23

Contents

DIAMOND LIGHT SOURCE ANNUAL REVIEW 2022/23

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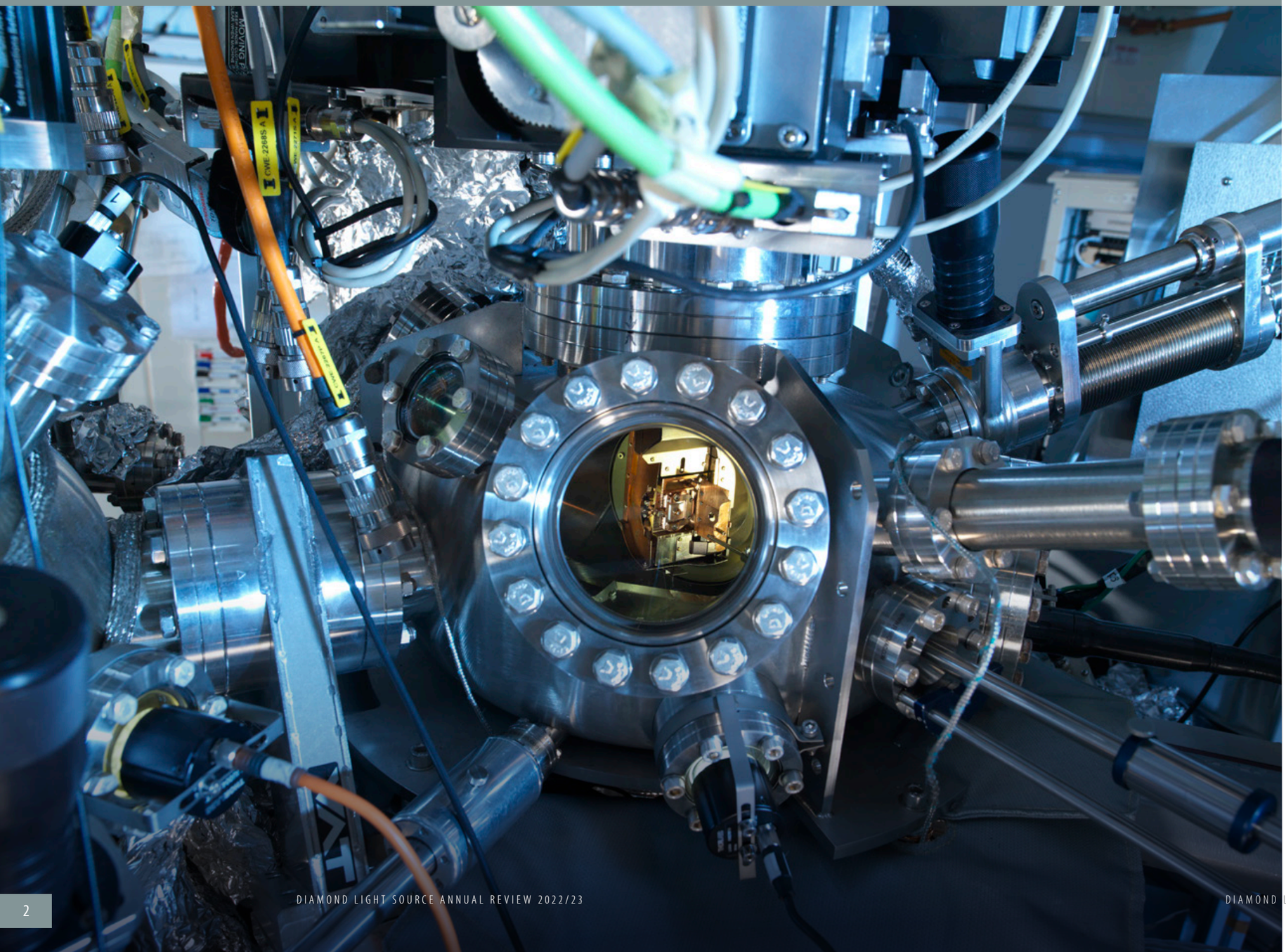


Foreword

As I enter my 9th year of involvement with Diamond, it remains a privilege to continue to be the Chair of the Board of such a world class research facility. The Board and I will continue to seek to support in all possible ways the Shareholders, the Executive and all the workforce in driving the organisation forward and maintaining our outstanding national and international reputation. In June 2022, we all welcomed the funding announcement confirming the first phase of investment for Diamond-II, reflecting the importance of this project to the UK and signalling a bright future for the organisation.

However, there continue to be challenges. In particular, recruiting and retaining appropriate talent against the background of pay constraint policies, inflation and a competitive jobs market is becoming a major issue.

This issue will remain at the forefront of the Board's priorities going forward and we will continue to share our concerns and argue our case at the highest levels with our funding and policy colleagues in UKRI, Wellcome and Government. With that assurance, let me end by extending on behalf of the Board our gratitude and admiration to all of you who contribute to the success of Diamond.

Professor Sir Adrian Smith
Chairman of the Board of Directors

CEO Welcome

2022 has been a year filled with many changes. As CEO, for an interim period, I took over from Professor Andrew Harrison last November to navigate the transition whilst Shareholders look for a long-term appointment. I have been on Diamonds Board as an Executive Director since April 2019.

Diamond has been delivering excellent science in collaboration with its user community, fostering knowledge that is addressing many of our global challenges.

Diamond now has 33 operational beamlines, which together with complementary facilities – eBIC, ePSIC, the MPL together with the XChem Fragment Screening service and the XFEL Hub - provide a truly integrated facility for virtually every field of science.

The switch from pandemic to endemic in the management of the coronavirus has meant a slowdown in our normal activities. The past year saw us increase our total of peer reviewed journal articles to some 12,384. This is a slower increase than expected as the effects of the lost measurements during the pandemic are starting to affect our numbers. Last year, there were 9,193 user visits to Diamond via academic access routes. In the last financial year, we received 1,906 proposals for experiments on beamlines and electron microscopes, which requested a total of 24,252 shifts. This resulted in 15,413 experimental shifts being awarded, spread across the 33 operational beamlines and seven of the electron microscopes. Within academia, 5,279 of our user visits were on-site visits, while the remaining 42% (3,914) were done via remote access.

We have seen reassuring signs of recovery with more beamtime to users being delivered but not yet to the full levels we reached prior the start of the pandemic. As new challenges emerge like the heightened threat of industrial action, the path to full recovery will take time. Recruitment and retention of our staff is now a major issue. With turnover levels increasing, I am working closely with our chairman to raise these issues at the highest levels with our shareholders. Like many other STEM institutes; we are faced with this issue, but we are working tirelessly to bring this issue to the attention of the UK Government to help us address this challenge.

In autumn 2022 we presented an update of our socioeconomic impact report at the International Conference of Research Infrastructures in Czech Republic. The report was initially published in May 2021 and undertaken by Technopolis together with representatives from our funding agencies. The report has become an influential piece of work within the research infrastructure community with many learning from its methodology and approach. With over 14,000 researchers interested in using our instruments, our position remains very strong given that Diamond has for the period 2007-2022 achieved a cumulative monetised impact of at least £2.6 billion whilst remaining extremely good value for money for the UK taxpayer who each contribute less than the cost of a cup of coffee each year. These tremendous benefits are a credit to our dedicated staff, contractors and agency workers who enable innovation, push the boundaries of what can be measured and offer excellent support and service to the science delivered internally as well as externally.

The financial year 2022-23 also marked our double anniversary – 20 years since the company was started and 15 years of research and innovation together with our user community. In broad terms, the public ultimately pay the taxes that underpin 86% of our funding. We celebrated in part with a large outdoor photo exhibition showcasing some of the beautiful imagery created over the years. We have started again to open our doors to the public, allowing them to see our incredible science and engineering, but more importantly, to experience the range of careers involved with Diamond's operation. We have welcomed over 80,000 visitors since opening in 2007, and it is always astounding how much interest the public show in our work, and humbling to think that many of the young people we are showing around the facility could soon be working here. Diamond is not only a visible landmark in the Oxfordshire landscape but has also become a cornerstone of the Harwell Campus with key investment being brought close to us for synergy.

The Diamond-II upgrade programme is an integrated upgrade of the synchrotron, beamlines and computational facilities, which is critical to maintaining our world-leading status. We further progressed with the first phase of funding allocated by UKRI and Science and Technology Facilities Council (STFC) and Wellcome in June 2022. A major milestone in securing full funding from the UK Government was achieved in November 2021 with approval of the Outline Business Case (OBC) by the then Department for Business, Energy and Industrial Strategy (BEIS) and Her Majesty's Treasury (HMT). This approval built on an early commitment of support from Wellcome for their funding share. This past year saw us deliver the Technical Design Report (TDR) for the machine, alongside Conceptual Design Reports (CDRs) for three flagship beamlines. As we put our finishing touches to the Full Business Case (FBC), which is due for review in June 2023 by the Programme Investment Committee at the newly formed Department for Science Innovation and Technology (DSIT), if approved, will move on to HM Treasury for final approval for release of full funding for the Programme. We are grateful to Dr Richard Walker, who took a lead as Interim Senior Responsible Owner for his leadership of the machine TDR and the FBC. This upgrade will increase electron beam energy from 3.0 to 3.5 GeV will provide up to a factor of 70 increase in brightness and coherence of Diamond's photon beams at the higher energies.

As I lead the organisation towards steps ever closer to its upgrade, I remain determined to see Diamond positioned at the forefront of scientific research in the UK, Europe and on a global stage. Science is ultimately a collaborative endeavour to better our lives, and in Diamond's case, the team remains focused on playing a key role in addressing 21st century global challenges.

Andrea Ward FCMA

CEO & Director of Finance and Corporate Services Diamond Light Source

"As I lead the organisation towards steps ever closer to its upgrade, I remain determined to see Diamond positioned at the forefront of scientific research in the UK, Europe and on a global stage."

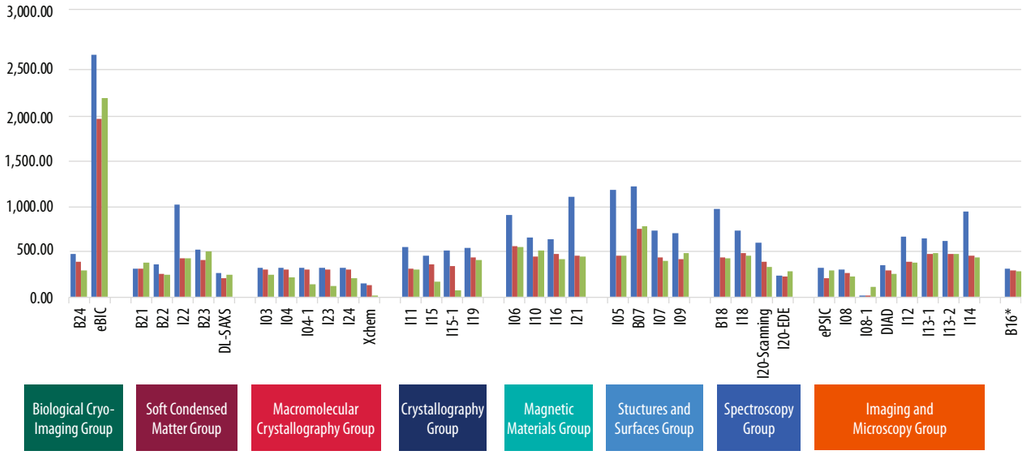
Artist's impression.

Key Facts and Figures

Facility usage

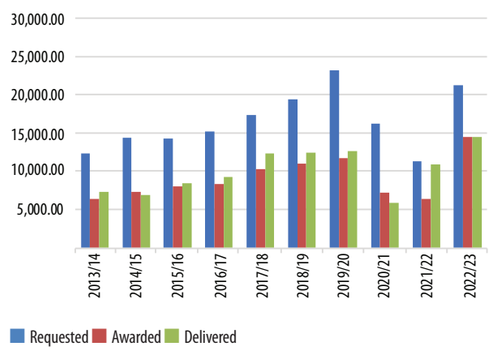
In our sixteenth year of operations (1st April 2022 to 31st March 2023), we received 1,906 proposals for experiments on our instruments via peer reviewed access routes, requesting a total of 24,252 shifts. After peer review, 1,420 proposals were awarded beamtime. This resulted in 16,478 experimental shifts being awarded across 33 beamlines and seven electron microscopes delivering time to academic users. We welcomed 5,279 onsite user visits from academia across all instruments, with an additional 3,914 remote user visits. The machine continues to perform to the highest standard with 97.3% uptime and 112 hours mean time between failures (MTBF).

User shifts requested, awarded and delivered by group, beamline and electron microscope 2021/22



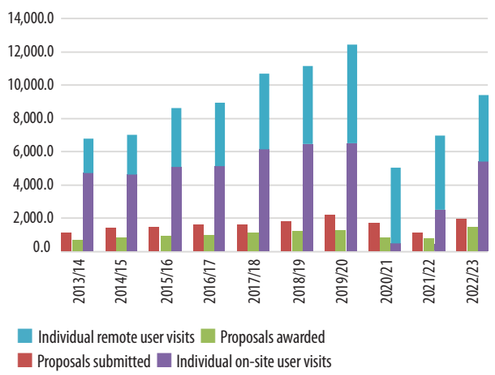
* B16 is the Test beamline, with 50% of beamtime for users. The rest is used for in-house developments for all beamlines.

Total user shifts requested, awarded and delivered



** also includes shifts requested awarded and delivered on labs and offline facilities*

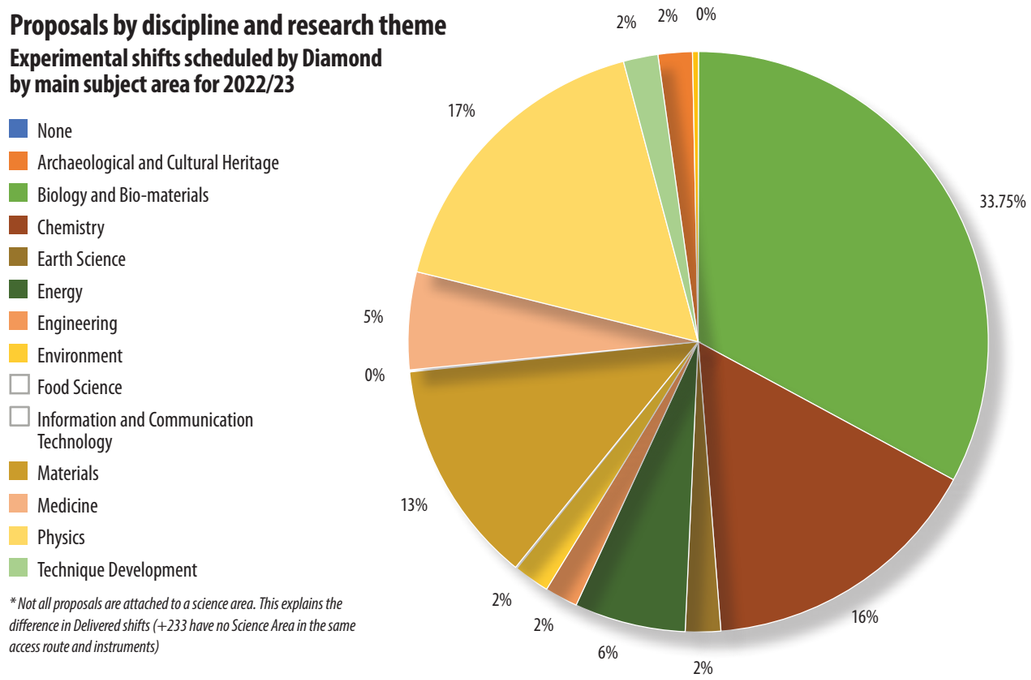
Total numbers of proposals and users per year



** Staff visits are now included for academic access routes, in-house research is still excluded*

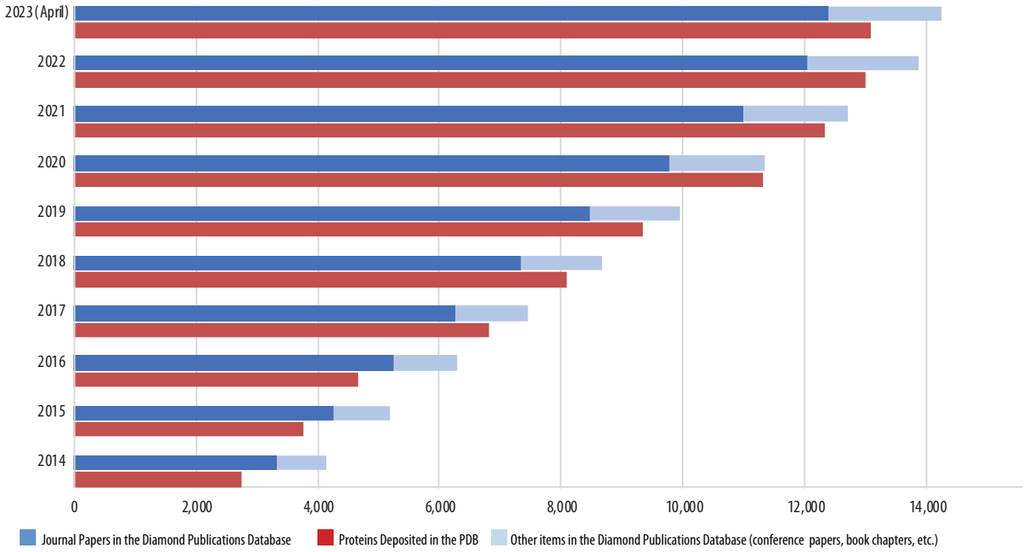
Proposals by discipline and research theme

Experimental shifts scheduled by Diamond by main subject area for 2022/23



** Not all proposals are attached to a science area. This explains the difference in Delivered shifts (+233 have no Science Area in the same access route and instruments)*

Cumulative number of items in Diamond Publications Database by our scientists and users and cumulative number of protein structures solved



Machine performance

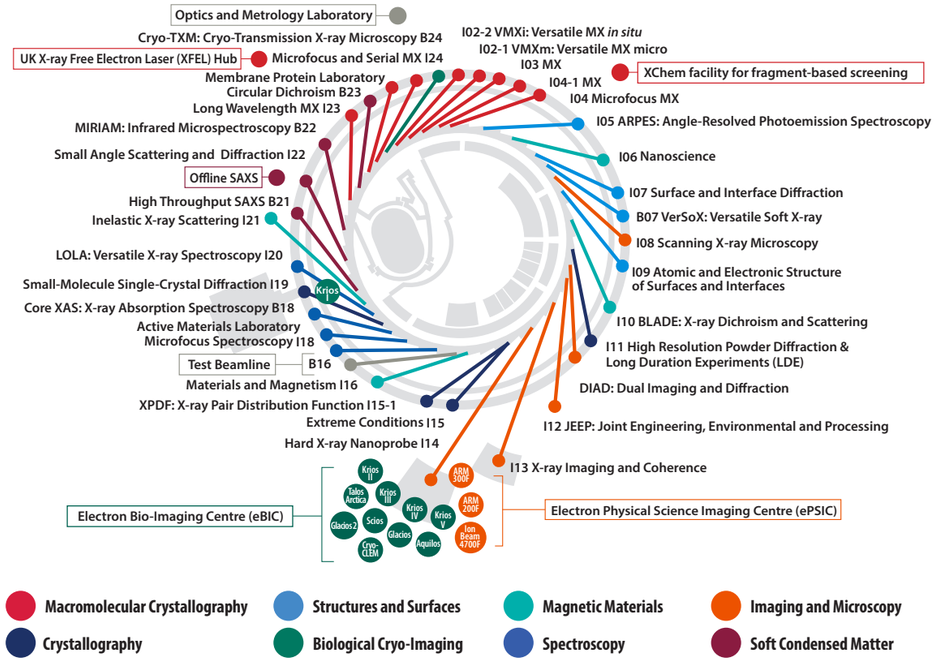
	2010/11	2011/12	2012/13	2013/14	2014/15	2015/16	2016/17	2017/18	2018/19	2019/20	2020/21	2021/22	2022/23
Total no. operational beamlines by end FY	19	20	22	24	25	26	28	31	32	32	33	33	33
Scheduled hours of machine operation	5808	6000	5832	5976	5808	5928	5688	6072	5904	5913	4345*	5396	6009
Scheduled hours of user operation	4728	5064	4872	5088	4944	5040	4584	5160	4992	4992	3445*	4532	5145
Machine uptime %	97.5	97.7	98.3	98.2	97.6	97.6	98.7	98.2	98.4	98.1	96.2	97.4	97.3
Mean time between failures (hours)	28.5	55.4	52.4	60.3	38.6	119.4	103.1	79.9	90.3	104.7	132	110	112

** hours reduced due to COVID*

Beamline Development and Technical Summary

In its sixteenth year of experiments, Diamond is now operating with 33 beamlines and eight electron microscopes dedicated for experiments. A further five instruments are available for experiment support and sample preparation. Six of the instruments specialise in life sciences and make up eBIC (electron Bio-Imaging Centre), with two provided for industry use in partnership with Thermo Fisher Scientific. Two of the electron microscopes are dedicated to advanced materials research and are supplied by Johnson Matthey and the University of Oxford. These, along with a further instrument for sample preparation, form ePSIC (electron Physical Science Imaging Centre) and are operated under strategic collaboration agreements to provide for substantial dedicated peer reviewed user access. Both eBIC and ePSIC are next to the Hard X-ray Nanoprobe beamline (I14). Along with eBIC and ePSIC, the UK X-ray Free Electron Laser (XFEL) Hub, the Membrane Protein Laboratory (MPL), the XChem fragrment screening facility and the Offline SAXS facility make up the complementary integrated facilities available at Diamond. For academic research, Diamond instruments (beamlines and microscopes) are free at the point of access through peer review. For proprietary research, access can be secured through Diamond's industry team.

The instruments and beamlines are organised into eight science groups as described below.



Macromolecular Crystallography **Structures and Surfaces** **Magnetic Materials** **Imaging and Microscopy**

Crystallography **Biological Cryo-Imaging** **Spectroscopy** **Soft Condensed Matter**

Electron Microscopes

Microscope	Main Capabilities	Accelerating Voltages	Operational Status
Titan Krios I	Cryo-EM, Cryo-ET	80, 120, 200, 300 kV	Operational since 2015
Titan Krios II	Cryo-EM, Cryo-ET	80, 120, 200, 300 kV	Operational since 2016
Titan Krios III	Cryo-EM, Cryo-ET	80, 120, 200, 300 kV	Operational since 2017
Titan Krios IV	Cryo-EM, Cryo-ET	80, 120, 200, 300 kV	Operational since 2017
Titan Krios V (Industrial)	Cryo-EM, Cryo-ET	80, 120, 200, 300 kV	Operational since 2018
Glacios (Industrial)	Cryo-EM, Cryo-ET	200 kV	Operational since 2019
Aquilos 2	Cryo-SEM, Cryo-FIB	3 to 30 kV	Operational since 2020
Leica cryo-CLEM	Cryo-CLEM	N/A	Optimisation
JEOL ARM200F	Atomic scale STEM imaging, EELS, EDX, electron diffraction	80, 200 kV	Operational since 2017
JEOL ARM300F	Atomic scale TEM and STEM imaging, electron diffraction, 4D-STEM, EDX	30, 60, 80, 160, 200, 300 kV	Operational since 2017
JEOL Ion Beam 4700F	SEM, FIB	1 to 30 kV	Operational since 2020

Diamond's beamlines: current operational status April 2022

Beamline Name and Number	Main Techniques	Energy / Wavelength Range	Status
I02-1 - Versatile MX micro (VMXm)	Micro- and nano-focus in vacuum cryo-macromolecular crystallography (VMXm)	7 - 28 keV	Optimisation
I02-2 - Versatile MX *in situ* (VMXi)	*In situ* microfocus macromolecular crystallography, Serial Synchrotron Crystallography	10 - 25 keV	Optimisation
I03 - MX	Macromolecular crystallography (MX), Multiwavelength Anomalous Diffraction (MAD)	5 - 25 keV	Operational
I04 - Microfocus MX	MX, MAD, variable and microfocus MX	6 - 18 keV	Operational
I04-1 - Monochromatic MX	MX, XChem fragment screening	13.53 keV (fixed wavelength)	Operational
I05 - ARPES	Angle-Resolved PhotoEmission Spectroscopy (ARPES) and nano-ARPES	18 - 240 eV; 500 eV	Operational
I06 - Nanoscience	X-ray Absorption Spectroscopy (XAS), X-ray photoemission microscopy and X-ray magnetic circular and linear dichroism	80eV - 2200eV	Operational
I07 - Surface and Interface Diffraction	Surface X-ray diffraction, Grazing Incidence X-ray Diffraction (GIXD), Grazing Incidence Small Angle X-ray Scattering (GISAXS), X-ray Reflectivity (XRR)	6 - 30 keV	Operational
B07 - VerSoX: Versatile Soft X-ray	Branch C: Ambient Pressure XPS and NEXAFS	110 - 2800 eV	Operational
	Branch B: NEXAFS and High-Throughput XPS	45 - 2200 eV	Operational
I08 - Scanning X-ray Microscopy	Scanning X-ray microscopy, NEXAFS/ XANES, X-ray fluorescence	I08 branch: 250 eV - 4.4 keV	Operational
		I08-1 - Soft and Tender X-ray Ptychography branch: 250 - 2000 eV	Operational
I09 - Atomic and Electronic Structure of Surfaces and Interfaces	XPS (including HAXPES), X-ray Standing Waves (XSW), Near Edge X-ray Absorption Fine Structure (NEXAFS), energy-scanned photoelectron diffraction	Hard X-rays: 2.1 - 18+ keV Soft X-rays: 0.1 - 2.1 keV (currently 0.1 - 1.9 keV)	Operational
I10 - BLADE: Beamline for Advanced Dichroism Experiments	Soft X-ray resonant scattering, XAS and X-ray magnetic circular and linear dichroism	Circular: 400-1600eV; Linear Horizontal: 250-1600eV; Linear Vertical: 480-1600eV	Operational
I11 - High Resolution Powder Diffraction	X-ray powder diffraction	7 - 25keV (1.7 - 0.5 - 2.1 Å)	Operational
DIAD: Dual Imaging and Diffraction	Simultaneous time-resolved X-ray imaging and X-ray powder diffraction	8 - 38 keV	Operational
I12 - JEEP: Joint Engineering, Environmental and Processing	Time-resolved imaging and tomography; 2D detector for time-resolved powder diffraction, single crystal diffraction and diffuse scattering; energy dispersive X-ray diffraction (EDXD); high-energy small angle X-ray scattering (limited capability)	53 keV - 150 keV monochromatic or continuous white beam	Operational
I13 - X-ray Imaging and Coherence	Phase contrast imaging, tomography, full-field microscopy (under commissioning), coherent diffraction and imaging (CXRD,CDI), ptychography and photocorrelation spectroscopy (XPCS) (under commissioning), innovative microscopy and imaging	Imaging branch: 8 - 30keV	Operational
		Coherence branch: 7 - 20keV	
I14 - Hard X-ray Nanoprobe	Nanofocus X-ray fluorescence (XRF), X-ray absorption spectroscopy (XAS), and transmission diffraction (XRD) mapping., differential phase contrast (DPC) imaging,), ptychography and tomography	5 - 23 keV	Operational
I15 - Extreme Conditions	Powder diffraction, single crystal diffraction	Monochromatic and focused 20 - 80 keV White beam	Operational
I15-1 - XPDF	X-ray Pair Distribution Function (XPDF)	40, 65, and 76 keV	Operational
I16 - Materials and Magnetism	Resonant and magnetic single crystal diffraction, fundamental X-ray physics	2.5 - 15 keV	Operational
B16 - Test beamline	Diffraction, imaging and tomography, topography, reflectometry	4 - 20 keV monochromatic focused 4 - 45 keV monochromatic unfocused White beam	Operational
I18 - Microfocus Spectroscopy	Microfocus X-ray Absorption Spectroscopy (XAS), X-ray fluorescence (XRF) and X-ray diffraction (XRD) mapping and tomography	2.05 - 20.5 keV	Operational
B18 - Core XAS	X-ray Absorption Spectroscopy (XAS)	2.05 - 35 keV	Operational
I19 - Small-Molecule Single-Crystal Diffraction	Small-molecule single-crystal diffraction	5 to 25 keV / 0.5 to 2.5 Å	Operational
I20 - LOLA: Versatile X-ray Spectroscopy	X-ray Absorption Spectroscopy (XAS), X-ray Emission Spectroscopy (XES) and Energy Dispersive EXAFS (EDE)	Dispersive branch: 6 - 26 keV	Operational
		Scanning branch: 4.5 - 20 keV	Operational
I21 - Inelastic X-ray Scattering	Resonant Inelastic X-ray Scattering (RIXS), X-ray Absorption Spectroscopy (XAS)	Currently 250 - 1500 eV (to be upgraded to 250 - 3000 eV)	Operational
B21 - High Throughput SAXS	BioSAXS, solution state small angle X-ray scattering	8 - 15 keV (set to 13.1 keV by default)	Operational
I22 - Small Angle Scattering and Diffraction	Small angle X-ray scattering and diffraction: SAXS, WAXS, USAXS, GISAXS. Micro-focus SAXS Tensor Tomography.	7 - 20 keV	Operational
DL-SAXS (Offline SAXS instrument)	SAXS/WAXS, GiSAXS/GiWAXS	9.2keV	Operational
B22 - MIRIAM: Multimode InfraRed Imaging And Mircrospectroscopy	FTIR microscopy & FPA imaging FTIR and THz spectroscopy NEW FTIR nanospectroscopy s-SNOM and AFM IR	microFTIR: 5,000-500cm^{1}(2-20µm) FTIR/THz:10,000-10cm^{1}(1-1000µm) nanoFTIR: 14000-800cm^{1}(2.5-12.5µm)	Operational Commissioning
I23 - Long Wavelength MX	Long wavelength macromolecular crystallography	2.1 - 11 keV (1.1 - 5.9 Å)	Operational
B23 - Circular Dichroism	Circular Dichroism (CD)	Module A: 125-500nm for CD Imaging at 50 µm spatial resolution, and 96-cell HTCD. Module B: 180-650nm for MMP Imaging at 50 µm spatial resolution.	Operational
I24 - Microfocus and Serial MX	MX, MAD, Serial Crystallography, high energy MX	7 - 30.0 keV	Operational
B24 - Cryo Transmission X-ray Microscopy (TXM)	Full field X-ray imaging	200eV - 2600eV	Operational

Macromolecular Crystallography Group

Dave Hall, Science Group Leader

Knowledge of the three-dimensional structure of biological macromolecules in concert with interacting partners, is essential to a deep understanding of their action. The users of the macromolecular crystallography (MX) facilities at Diamond (www.diamond.ac.uk/Instruments/Mx.html) wish to understand biological nature and function and the many and varied tools available to them through Diamond's MX group add significant value to their investigations.

Scientists can obtain high resolution structures rapidly, and automatically, across multiple X-ray beamlines. The widest range of biologically relevant metals in proteins and nucleotides can be identified and located and additionally these can be used to solve their structures where needed. Drug discovery programmes can take advantage of the fragment based screening platform provided by the XChem facility. For users with an interest in membrane protein biology a research and training facility is available through the Wellcome funded Membrane Protein Laboratory. Room temperature studies of samples *in situ* from crystallisation plates or via serial synchrotron crystallography (SSX) including time resolved studies are well supported and UK SSX users of international XFEL facilities can gain further assistance with their experiments and travel assistance via the XFEL-Hub team. For the most challenging of micro-crystals the group has several facilities available and is pushing the extremes of what is feasible with X-rays and electrons with the latest instruments in development.

For example, for micro-crystals excellent progress continues to be made on the electron diffraction HeXI project with successful external scientific reviews completed and a full beamline-grade Technical Design Review for phase 1 recently finalised. The instrument construction will begin imminently, and results will be used to inform the conceptual design review of the high-energy phase 2 instrument, which is scheduled to be completed by the summer. The commissioning of the newly installed hybrid pixel Quantum Detectors Merlin Quad EM 4S on the offline TF30 electron microscopes continues and recently acquired datasets are currently undergoing analysis.

The HeXI project is closely linked to the sub-micron focus beamline VMXm which this year has been building its user programme via a commissioning call. Here, both academic and industrial users have been exploring what this unique beamline can achieve with the smallest of samples, including this year the first structures to have been experimentally phased at the beamline. Studies have included both biological macromolecules and small molecules which present different challenges to the experimental set-up. Interacting with scientists from a broad range of disciplines and varied samples is a rewarding and fruitful exercise for the beamline team through this commissioning phase to bring the beamline to a wider audience.

I24 provides a dual offering of 'standard' MX and serial synchrotron crystallography (SSX) using a microbeam. A fully flexible beam – in both size and energy – is available with efficient data collection at high energies (> 20 keV) possible with the CdTe Eiger detector. New endstation hardware has been developed to ease switching between experimental modes. For dynamic experiments, a range of approaches are available using either LEDs, the PORTO laser system, or *in situ* picodroplet ejection. Complementary spectroscopic approaches have been used by several groups and new improved hardware for *in situ* validation of dynamic experiments is under development. Real-time assessment of serial data for users has become simpler with data collections recorded in ISPyB and automated pipelines in place providing indexing and integration of data in addition to live hit-rates.

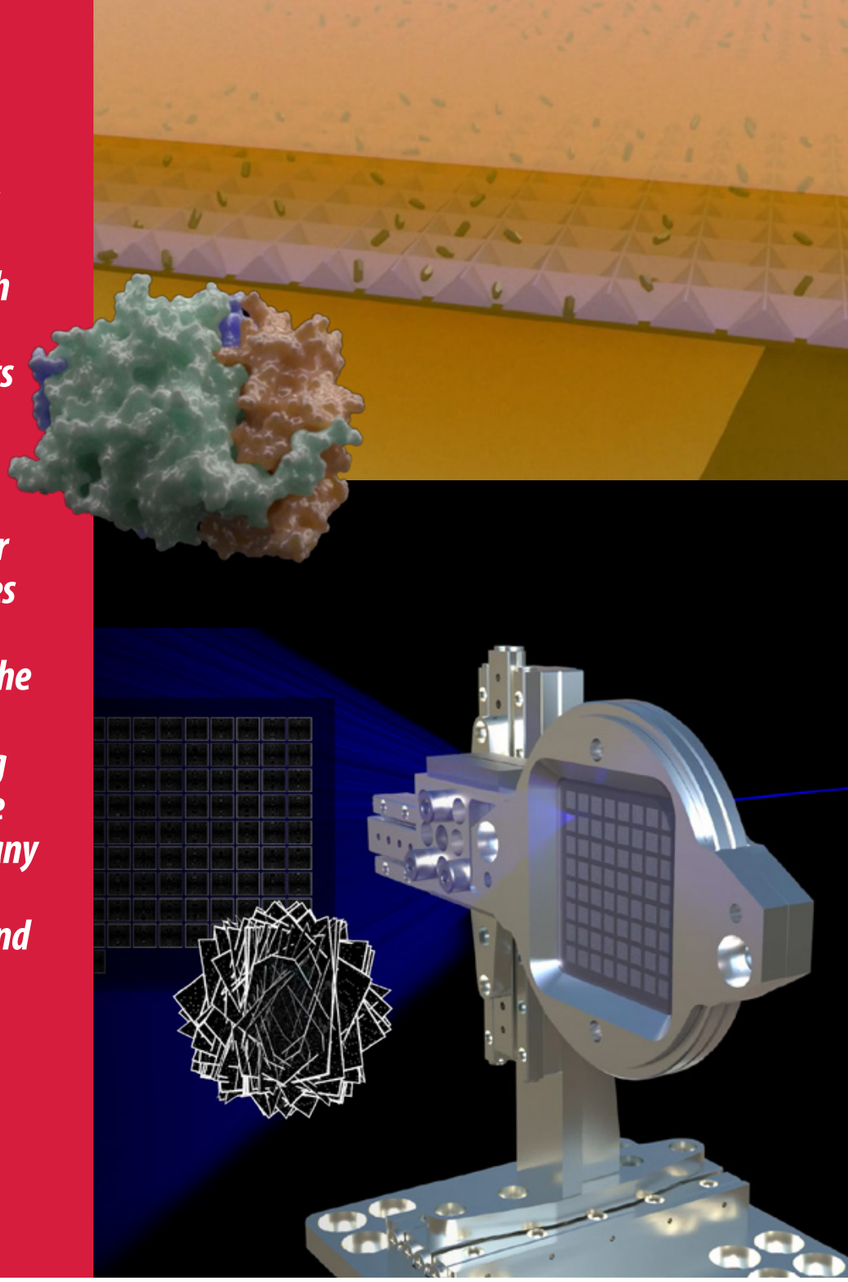
VMXi is now available to users through MX BAG and rapid access proposals. The beamline offers highly automated and remote data collection from crystals as small as 10 µm *in situ* within crystallisation plates. Identification and selection of crystals from optical images has recently been automated using a machine learning tool and merged multi-crystal datasets (via Dials Multiplex) are automatically available to users within ISPyB without manual intervention. Typical room temperature datasets use 3-20 crystals with structures determined to as high as 1.5 Å resolution. Grid scans to identify crystalline material within drops are available and a serial crystallography capability using a tape drive coupled with X-ray emission spectroscopy is under development in partnership with the UK XFEL Hub.

A variety of studies have been published exploiting the unique wavelength range offered by the long-wavelength MX beamline I23. Significant effort has been dedicated to streamline the sample loading into vacuum and automate the sample changes inside the large vacuum end station. Experiments to wavelengths as low as 5 Å can now be routinely performed. Protocols to deal with the increased absorption at these very long wavelengths are being actively pursued. New software for analytical absorption corrections based on 3D models of the samples obtained from X-ray tomography are under development. An alternative approach is to use a high-power laser to remove the non-diffracting materials around the crystals to facilitate absorption corrections. First successful tests have been performed and over the next months we anticipate commissioning a dedicated system to become part of the I23 sample workflows for very long wavelength experiments.

I04 has implemented further improvements of the dose tool for data collection and the use of dialling a target dose instead of an exposure time has been taken up by more users and has been well received with very positive feedback. I04 staff actively promote this and are providing in-person training on the beamline on this and other aspects of data collection. The dose information is now also displayed in SynchWeb and this allows the experimenter and I04 staff to assess if a suitable dose has been used based on the scientific aim of the experiment. This also allows for a better comparison between data sets. The use of the dose-aware data collection is even more important since a new X-ray source (CPMU insertion device) was installed on I04 in the June 2022 shutdown. This resulted in 2-7 times higher flux depending on the energy and in turn results in a much more intense microfocus beam. As a result, the detector can now be used routinely at its fastest acquisition rate of 500 Hz, increasing sample throughput, which also greatly benefits Unattended Data Collection (UDC). Furthermore, this also enables tackling more challenging samples in the microfocus regime and opens up the possibility to extend the beamline energy range to higher energies.

I03 has continued to develop and offer UDC as its main mode of operation working closely with beamlines I04 and I04-1 to widen the usage of automated collection via UDC at Diamond. The ability to screen crystals for diffraction resolution prior to collecting data for the user aims has broadened the appeal and uptake of UDC by users. This allows users to select for collection if a sample meets a particular resolution requirement or to screen and collect the best of a

"This year has seen the return of users to site not only for beamtime but also for training with in particular many early career scientists having missed out on the opportunity to visit large scale facilities during their training programmes due to COVID-19 working protocols. The beamline teams are actively encouraging users to come on-site to experience the many changes that have been made during and after the COVID-19 pandemic"



range of samples. The I03 team together with experts from the SSCC group are using I03 to testbed the next generation data acquisition and analysis systems with UDC. Here new features have been developed for crystal centering using "3D grid scans" which greatly speed up and improve centering accuracy as well as open up the potential to rapidly identify multiple crystals within a single sample holder amongst many other opportunities. This new software is being developed ultimately for Diamond-II beamlines however MX users will see benefits and improvements to their future experiments before the Diamond-II upgrade with I03 at the forefront of delivering these new features.

UDC has also been rolled out to beamline I04-1 this year where it is now used for all of the data collections for XChem – the fragment based drug discovery laboratory. There have been increases in laboratory capabilities this year to cope with an increase in demand from both academic and industrial users coupled with requirements to deliver for multiple international grant funded collaborations including most recently the AI-driven Structure-enabled Antiviral Platform (ASAP, NIH funded) which aims to discover and develop accessible and affordable oral antivirals against COVID-19 and future pandemics. In parallel the team have been working on the flagship beamline K04 as part of the Diamond-II upgrade with the conceptual design report finalised this year.

This year has seen the return of users to site not only for beamtime but also for training with in particular many early career scientists having missed out on the opportunity to visit large scale facilities during their training programmes due to COVID-19 working protocols. The beamline teams are actively encouraging users to come on-site to experience the many changes that have been made during and after the COVID-19 pandemic and we have run our first on-site BAG trainings and Diamond-CCP4 workshop for three years. These on-site visits have provided invaluable opportunities for two-way exchange that have been much missed over recent years.

Characterisation of the enzymatic degradation of PET plastic as found in plastic bottles

Earth Sciences & Environment – Biotechnology – Pollution – Catalysis – Chemistry – Structural Biology – Materials Science – Engineering & Technology – Biophysics – Polymer Science – Life Sciences & Biotech

Plastic products may have revolutionised the world, but their poor degradability is causing major pollution problems. Poly(ethylene terephthalate) (PET) is one of the most abundant plastics used in polyester textiles and in packaging for food and drinks. PET's water-repellent properties make it a good choice for drinks bottles. However, they also make discarded PET bottles highly resistant to breaking down in the natural environment. Remaining intact for hundreds of years, PET waste will accumulate unless we can find a way to deal with it.

A possible solution presented itself in 2018, when an international team of researchers announced the discovery of a bacterium with the amazing ability to use plastic as an energy source. *Ideonella sakaiensis,* initially found feeding on waste from an industrial PET recycling facility in Japan, degrades PET from plastic bottles into its building blocks. Central to this ability is the bacterium's production of a PET-digesting enzyme called PETase. PETase is a mesophilic enzyme, meaning that it is only stable at moderate temperatures (30°C). However, PET degradation is better at higher temperatures. Therefore, a more thermostable enzyme is desirable.

The team's ongoing research has produced and identified PETase variants, including a double amino acid mutant (mutating residue 159 from tryptophan to histidine and 238 from serine to phenylalanine) with improved PET degradation relative to the wild-type PETase. In this work, they used Diamond's I03 beamline to examine the structural basis of the double mutant's enhanced PET degradation in greater detail. The 1.45 Å resolution data set they collected for the double mutant allowed them to determine its atomic protein structure. They also used differential scanning calorimetry to determine the melting temperatures of the enzymes and high-performance liquid chromatography to quantify the products in biochemical assays.

Their results show that the double mutant PETase narrows the active site cleft and has a 10°C higher melting temperature than the wild-type enzyme. Furthermore, while the activity of the wild-type enzymes drops after 48 hours, the double mutant PETase is active at 40°C for more than a week. However, the wild-type enzyme is more active at 30°C than the double mutant and is also more active at 40°C within the first two days.

The past decade has seen incredible progress in identifying, characterising and engineering PET hydrolases, including the PETase from *Ideonella sakaiensis* featured in this study. However, engineering the enzyme is just one of the tools in the toolkit for improving efficiency. Reaction optimisation and process design tuned to the characteristics of the waste stream may also prove critical in developing an efficient enzymatic recycling process. Strategies for implementing and scaling-up enzymatic recycling technologies are on the horizon.

Related publication:
Erickson, E. et al. Comparative performance of PETase as a function of reaction conditions, substrate properties, and product accumulation. *ChemSusChem* **15**, (2022). DOI: 10.1002/cssc.202101932

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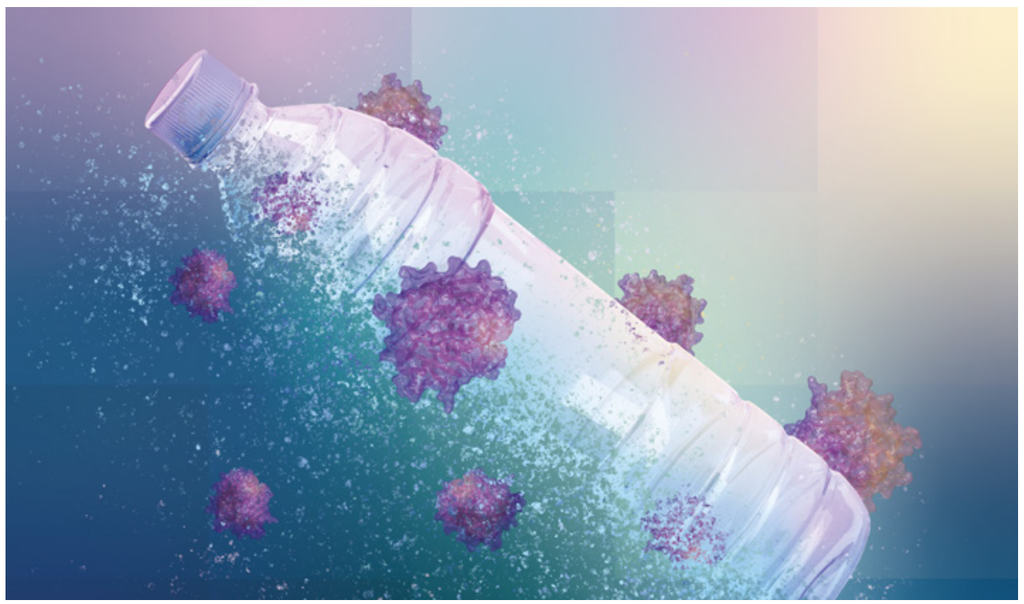

Artist's interpretation of a plastic PET bottle degradation by enzyme PETase.

Understanding the biochemistry of gut bacteria

Biochemistry – Chemistry – Genetics – Structural Biology – Life Sciences & Biotech

Lipids are vital components of all cells, forming the main component of cell membranes. Many different lipids are found in membranes, with a wide range of functions beyond their primary role in cellular compartmentalisation. Inositol is a carbocyclic sugar that is a key player in eukaryotic cells and forms the polar head group of inositol lipids. Inositol lipids are not major components of eukaryotic cell membranes, but they play important roles in cell division and signalling between cells. While inositol lipids are widely distributed across eukaryotes, little is known of their role and structure in bacteria.

An international team of researchers focused on understanding how different lipids are synthesised in bacteria investigated how the dominant gut microbe *Bacteroides thetaiotaomicron* makes inositol lipids. *B. thetaiotaomicron* is an important member of the human gut microbiome and can use a wide range of dietary carbohydrates as carbon sources.

The team is interested in how these bacteria interact with the host through various signalling molecules, including lipids. For this research, they used a range of techniques, including lipid analysis and biochemical characterisation of enzymes combined with structural biology. Diamond's I04 Macromolecular Crystallography beamline is a versatile facility for macromolecular crystallography, with the ability to focus the beam on very small crystals and access different X-ray energies. The ability to adjust the beam size to the sample size in order to optimize signal to noise, combined with a very stable beam, allowed the researchers to obtain very high quality data that permitted structure solution and the generation of a high resolution model of one of the key enzymes in this pathway, the myo-inositol phosphate synthase.

After identifying the genes responsible for *B. thetaiotaomicron* inositol lipid, the researchers deleted each of these from the genome to see their effect on the production of the inositol lipids. They identified the different lipids produced in these genomic deletion strains using mass spectrometry. These results allowed them to determine the order in which different components of the final inositol lipid are combined.

B. thetaiotaomicron bacteria with genomic deletions in the inositol lipid synthesis genes are less able to survive in their host. Although we do not know exactly why this is the case, it may be due to changes in the cell membrane and associated protective polysaccharide capsule around the cells that makes the cells more susceptible to antimicrobial compounds produced by the host, or change how the cells are seen by the host's immune system.

Changes in the composition of the gut microbiome can affect host health. By developing an understanding of the biochemistry of bacteria in the gut, we can gain knowledge of the roles they play in various diseases, such as inflammatory bowel diseases. The role of inositol lipids in eukaryotic signalling and cellular homeostasis is well established, and the fact that they are produced by important members of the human microbiome has implications for communication between these bacteria and their hosts. As we learn more about the lipids made by bacteria and how they are made, we can investigate how they use them to manipulate their host to survive and thrive.

Related publication:
Heaver, SL, *et al.* Characterization of inositol lipid metabolism in gut-associated Bacteroidetes. *Nature Microbiology* **7**, 986–1000 (2022). DOI: 10.1038/s41564-022-01152-6

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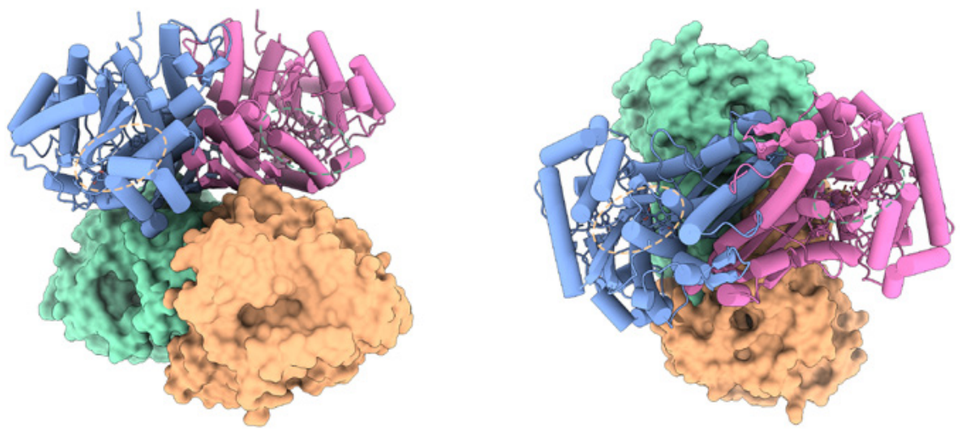

Structure of the B. thetaiotaomicron myo-inositol phosphate synthase enzyme. The enzyme adopts a structure with four identical subunits arranged as a dimer of dimers. Two subunits are shown with their molecular surfaces, and two with secondary structure elements displayed as tubes and arrows for alpha helices and beta-strands respectively. The NAD^+ cofactor, that participates in the isomerisation reaction to produce inositol-3-phosphate from glucose-6-phosphate, is highlighted in the structure and shown as sticks buried in the active site of the structure.

Fragment screening offers new leads in the search for Alzheimer's treatments

Neurodegenerative Diseases – Non-Communicable Diseases – Health & Wellbeing – Biochemistry – Neurology – Chemistry – Structural Biology – Organic Chemistry – Drug Discovery – Life Sciences & Biotech

In the nervous system, Wnt signalling is important for neuronal differentiation, development, and stem cell maintenance. Overall, neuronal Wnt signalling tends to decrease with ageing. At the same time, the Wnt inhibiting molecules, such as Notum, may increase, resulting in a reduction of neurogenesis and contributing to neurodegenerative diseases, such as Alzheimer's disease.

Notum has recently been identified as a negative regulator of Wnt signalling through the removal of an essential palmitoleate group from Wnt proteins, thus deactivating Wnts. Notum inhibitors could make more active Wnt protein available for signalling, thus offering therapeutic benefits for Wnt insufficient pathological states, such as Alzheimer's disease. However, some previously discovered Notum inhibitors cannot penetrate the blood-brain barrier, so researchers are engaged in crystallographic fragment screening for novel Notum inhibitors.

They carried out these experiments at Diamond's XChem platform. Built around MX beamline I04-1, XChem specialises in screening fragments directly in crystals by X-ray crystallography, using synthesis-aligned fragment libraries. With the sample preparation laboratory lab34 co-located with the beamline, it offers a highly streamlined process allowing up to 1,000 compounds to be screened individually in less than a week, including ~24 hours of unattended beamtime. The process covers soaking, harvesting, automatic data collection, and data analysis, and tailored software and automated systems enable researchers to record and track data seamlessly from initial crystal cultivation to data analysis.

Fragment screening has become increasingly popular over the last decade as it promises a step-change in early drug discovery process. It provides valuable and cost-effective insights for rational drug design, enabling scientists to identify high-quality lead candidates in the early stages of discovery. Rather than focusing on a few large complex compounds, it explores a larger part of the existing chemical space from the drug targets of interest, providing new entry routes for developing lead compounds.

This research involved soaking over a thousand compounds into Notum crystals and collecting X-ray diffraction data. After analysing 768 datasets, they found 59 compounds that bind to the Notum enzyme pocket with different potencies. Six hits were chosen for further development, and one (1-phenyl-1,2,3-triazole) has shown promising properties and the capability to penetrate the brain.

The lead compound, 1-phenyl-1,2,3-triazole, is now being used to assess when and in which tissues blocking the action of Notum could have beneficial effects by re-balancing the level of Wnt signalling.

Related publication:
Willis, NJ. *et al.* Design of a potent, selective, and brain-penetrant inhibitor of Wnt-deactivating enzyme Notum by optimization of a crystallographic fragment hit. *Journal of Medicinal Chemistry* **65**, 7212-7230 (2022). DOI: 10.1021/acs.jmedchem.2c00162.

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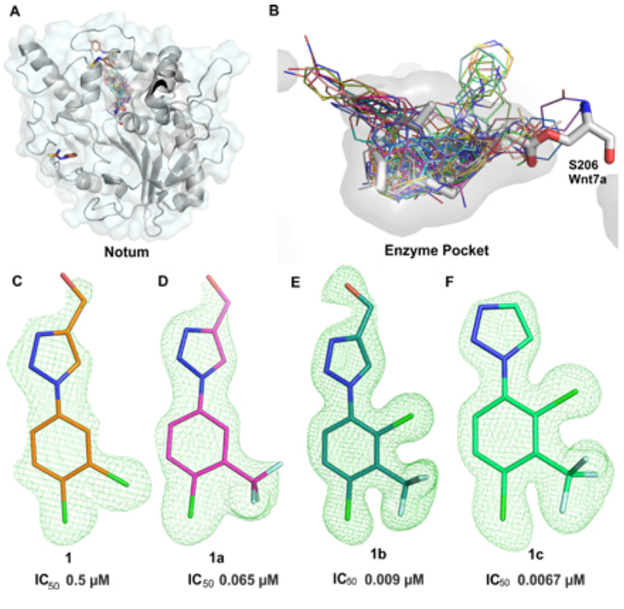

The Notum structure and the fragment hits within the enzyme pocket. (A) Notum structure shown as cartoon and fragment hits from complex structures were superimposed alongside the natural substrate (PAM)-bound structure. (B) Close-up view of the enzyme pocket hits. (C–F) The electron density maps for the fragment 1 and its derivatives with their potencies. 1c shows lead like properties with good brain penetrating capability.

Structural studies of the enhanced binding affinity of therapeutic nucleic acids to proteins

Health & Wellbeing – Biochemistry – Chemistry – Structural Biology – Drug Discovery – Life Sciences & Biotech

Introducing phosphorothioate (PS) linkages to the backbone of therapeutic nucleic acids significantly increases their stability and potency. The phosphorothioate backbone is the most widely used modification in therapeutic nucleic acids, including antisense oligonucleotides (ASOs). This modification involves a replacement of one of the two oxygen atoms in the repeating phosphate groups of the DNA with sulphur. PS-modified nucleic acids show improved properties, such as metabolic stability from nuclease-mediated degradation.

One of the hallmarks of PS ASOs is enhanced interactions with cellular proteins. This property, on the one hand, facilitates cellular uptake of nucleic acid drugs and their cell retention. However, on the other hand, it might contribute to the cytotoxic properties of the drug molecule. The molecular mechanisms of interactions between PS nucleic acids and proteins have not been fully established.

To better understand how PS ASOs interact with cellular proteins, researchers solved two crystal structures of PS ASO bound to annexin A2 (AnxA2), a calcium-binding protein previously implicated in the release of PS ASOs from endo-lysosomal compartments. The high quality of their sample crystals allowed them to use a unique experimental setup at the I23 beamline to perform long-wavelength X-ray diffraction experiments. These experiments led to precise localisation of the sulphur atoms in the structure and allowed the identification of PS stereoisomers in the DNA bound to the protein.

Their results unambiguously confirmed, for the first time, that van der Waals contacts between the sulphur atom and hydrophobic parts of arginine and lysine side chains are the driving force for enhanced interaction of PS ASO with proteins. Interestingly, stereoisomer preference at a given phosphorothioate in the DNA oligonucleotide is determined by the hydrophobic environment around the PS linkage coming not only from the protein but also from adjacent structural features within the ASO such as 5-Methyl groups on cytosine nucleobases.

Overall, their results provide valuable insights into the general mechanism of the enhanced binding of PS ASOs to cellular proteins and indicate that the interaction between PS linkages and lysine and arginine residues is a general phenomenon that is observed not only for nucleic acid-binding proteins but may also account for the association of ASO with proteins that are not known to bind DNA. This work provides information that will be instrumental in the rational design of improved nucleic acid-based drugs.

Related publication:
Hyjek-Składanowska M. *et al*. Structures of annexin A2-PS DNA complexes show dominance of hydrophobic interactions in phosphorothioate binding. *Nucleic Acids Research* **51,** 1409–1423 (2023). DOI: 10.1093/nar/gkac774

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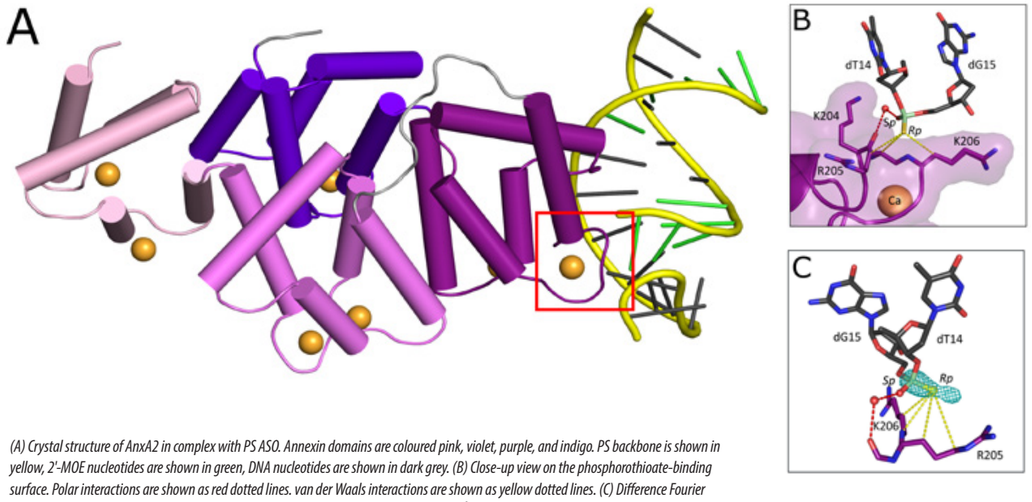

(A) Crystal structure of AnxA2 in complex with PS ASO. Annexin domains are coloured pink, violet, purple, and indigo. PS backbone is shown in yellow, 2'-MOE nucleotides are shown in green, DNA nucleotides are shown in dark grey. (B) Close-up view on the phosphorothioate-binding surface. Polar interactions are shown as red dotted lines. van der Waals interactions are shown as yellow dotted lines. (C) Difference Fourier anomalous map calculated based on long-wavelength X-ray diffraction data (λ = 2.7552 Å), shown as teal mesh. The preferred occupancy of Rp PS stereoisomer is facilitated by the hydrophobic interactions with surrounding amino acids.

Freeze-frame views of proteins in motion: how do acid and alkaline conditions alter the structure of a fluorescent protein?

Physical Chemistry – Biochemistry – Chemistry – Structural Biology – Life Sciences & Biotech

Proteins are the building blocks of life – our entire body is made from them, including structural proteins, enzymes, hormones and antibodies. X-ray crystallography is a well-established technique for determining the 3D structure of a protein crystal, which helps us to understand how proteins function. Serial Synchrotron Crystallography (SSX) is an emerging method that can capture a series of snapshots of proteins during motion and piece them together, like a stop-motion movie, to understand the critical steps and components in the protein's activity. This allows movies of protein motion to be captured on timescales spanning femtoseconds to seconds with resolution high enough to observe the movements of single atoms in the protein structure.

A wide array of reversibly photoswitchable fluorescent proteins (rsFPs) have been developed and are used across microscopy in biological sciences to track cell behaviour, develop bioelectronics and image living things. A key aspect of reversibly switchable fluorescent proteins is their ability to switch 'on' and 'off'. However, in low pH (acidic) conditions, this switching technique is poorly understood.

A team of researchers from Imperial College London conducted experiments to increase our understanding of the switching reactions in these proteins at low pH and compare it to neutral pH to see if there were differences in switching mechanisms.

Diamond's I24 beamline provided a bespoke set-up to capture freeze frames of the protein in motion, which the researchers combined with infrared spectroscopy measurements made in their own lab. Obtaining the structure at specific pH levels and illumination conditions allowed them to directly correlate particular structures to the infrared signals they measured. Using these techniques, the team discovered that at acidic pH, the photo-switching behaviour of their protein followed a different cycle to that at an alkaline pH. They also made the first observation of switching to a new chromophore charge state.

Experiments like this one demonstrate that the combination of synchrotron microfocus beams and serial crystallography is a powerful one. Working together, Diamond and Imperial were able to develop existing techniques into a new set of tools to investigate a particular biological problem.

Their results will directly inform the design of new reversibly switchable fluorescent proteins, especially with red-shifted emission spectra. It could also form the basis for new ultrafast measurements to study pathways of the photoexcitation in these samples.

Related publication:
Baxter, JM. *et al.* Observation of cation chromophore photoisomerization of a fluorescent protein using millisecond synchrotron serial crystallography and infrared vibrational and visible spectroscopy. *The Journal of Physical Chemistry B* **126,** 45 9288-9296 (2022). DOI:10.1021/acs.jpcb.2c06780.

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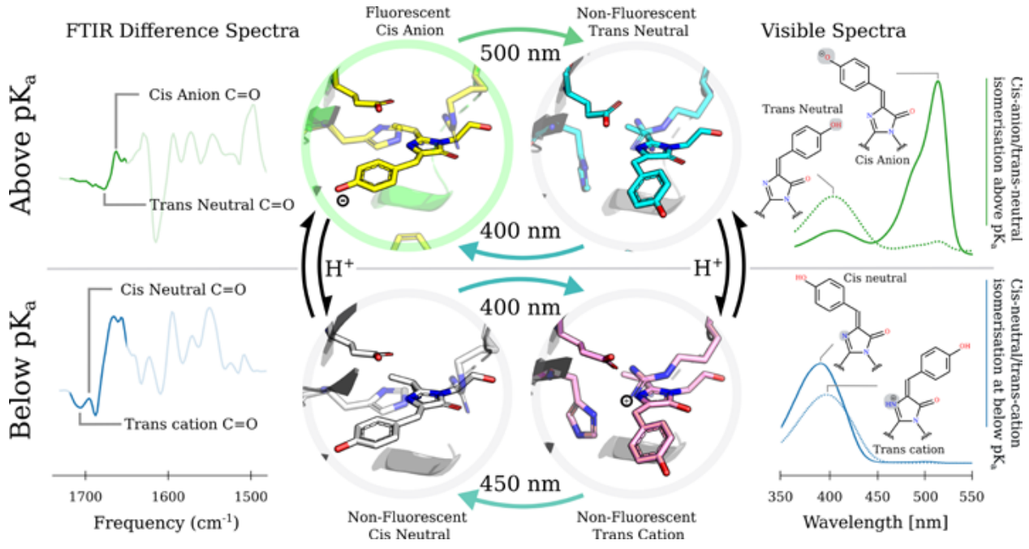

At pH below the chomophore pKa, the photocycle of an engineered fluorescent protein includes the first observation of photoisomerization of the cation state.

xia2.multiplex - a new pipeline for multi-crystal data analysis

Pathogens – Infectious Diseases – Health & Wellbeing – Information & Communication Technologies – Structural Biology – Data processing – Life Sciences & Biotech

A team of scientists at Diamond has developed a new program, xia2.multiplex, to facilitate and help to optimise the scaling and merging of multiple data sets.

At the very beginning of macromolecular crystallography, structures were obtained using room temperature data collection on weak (in comparison to synchrotrons) laboratory X-ray sources, and it was common to merge together data from multiple crystals to obtain a complete data set. With the increasing availability of synchrotron sources with ever more intense beams in the 1990s, radiation damage (i.e. the maximum X-ray dose that a crystal can tolerate) was becoming a limiting problem, leading to the popularisation of "cryogenic" data collection. Such flash-cooling of crystals to around 100 K significantly extends the crystal lifetime in the X-ray beam, thereby increasing the quantity of data that can be collected per crystal. As a result, most macromolecular structures obtained over the last 20-30 years were solved using data from a single crystal under cryogenic conditions.

Simultaneously, the development of highly-automated data analysis pipelines such as xia2 and fast_dp has made macromolecular crystallography more accessible to structural biologists, enabling more efficient use of beamtime for routine experiments.

However, while undoubtedly successful, cryocooling has a number of limitations. In recent years, there has been an increasing awareness that cryocooling can "hide" biologically significant structural features. So there has been interest in collecting data under more physiologically-relevant room temperature conditions (with the trade off that significantly less data can be obtained from a single crystal). Certain classes of macromolecular crystals, such as viruses, can suffer damage on cryocooling, necessitating room temperature data collection for improved data quality.

In addition, many scientifically important targets, such as membrane proteins and viruses, frequently yield small, weakly diffracting microcrystals. Improvements in beamline (more intense, microfocus beams) and detector (speed and sensitivity) technology and experimental techniques have made collecting data from such crystals more tractable. However, data processing remained challenging compared to the user experience for more routine experiments and multiple crystals are often required even at 100 K.

Combining incomplete datasets from multiple crystals poses several challenges. Firstly, determining an overall symmetry can be challenging when combining sparse datasets of varying quality. This problem is addressed by a new approach to symmetry determination implemented in the Diamond developed software dials.cosym and used by xia2.multiplex.

Other challenges in multi-crystal data collections include the identification and rejection of "non-isomorphous" or poor-quality datasets from particular crystals and the assessment of the levels of possible radiation damage, particularly for room temperature datasets.

Whilst this software development applies to all MX beamlines, it is particularly relevant to I24, VMXi and VMXm, where room temperature experiments or data collections on micro-crystals or room temperature crystals within their crystallisation droplet typically necessitate combining data from multiple crystals.

The research team has demonstrated that xia2.multiplex can be used to combine multi-crystal datasets. Its implementation within the wider MX data analysis pipelines makes it readily available to MX users at Diamond, providing them with timely feedback on multi-crystal experiments. xia2.multiplex can be applied to a wide variety of multi-crystal datasets, from multi-crystal phasing experiments on I23 to room temperature *in situ* data collections on I24 and VMXi. It is expected to play a critical role in future research investigating room temperature fragment screening on VMXi as part of the EU-funded "Fragment-Screen" project.

https://instruct-eric.org/news/fragment-screen-a-new-european-project-coordinated-by-instruct-eric/

Related publication:
Gildea, RJ. *et al.* xia2. multiplex: a multi-crystal data-analysis pipeline. *Acta Crystallographica Section D: Structural Biology* **78**, 6: 752-769 (2022). DOI 10.1107/S2059798322004399

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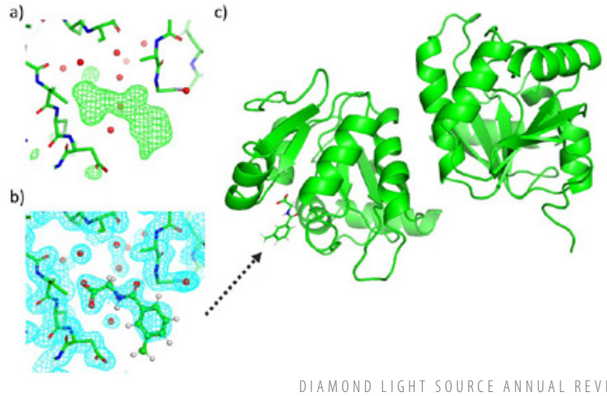

Room temperature ligand binding results using data from 8 crystals of SARS CoV2 macrodomain 1 (MAC1) collected at VMXi and merged together with xia2.multiplex. a) Evidence at the ligand binding site for the presence of the ligand (green mesh); b) Ligand included within the structure to show interactions with the protein; C) overall structure of the protein with the ligand binding site shown.

Biological Cryo-Imaging Group

Martin Walsh, Science Group Leader

The Biological Cryo-imaging Group brings together dedicated facilities for cryo-electron microscopy (cryo-EM), X-ray and super resolution light microscopies at Diamond Light Source. The electron Bioimaging Centre (eBIC) is the national centre for cryo-EM in the UK and provides a range of capabilities and supporting facilities for single particle cryo-EM, cryo-Electron Tomography (cryo-ET) and Correlative Light and Electron Microscopy (CLEM). Beamline B24 hosts a full field cryo-transmission X-ray microscope dedicated to biological X-ray imaging and has also established cryo super-resolution fluorescence microscopy, which is a joint venture between Diamond and the University of Oxford. It provides a unique platform for correlative light and X-ray microscopy, and cryo-EM.

The Biological Cryo-Imaging group continues to grow with enhancements to established instruments and new capabilities. At eBIC, there is a long list, but highlights include: 1) Roll-out of an external commissioning user program to access the correlative workflows as part of our cryo-ET access, through the use of the Leica cryo-CLEM, with first users taking advantage of this access route in 2022; 2) Access to semi-automated cryoFIB milling via the Aquilos-2 cryoFIB/SEM dual beam microscope is now fully integrated into the BAG access system. The Aquilos-2 will be further upgraded in June 2023 with the integration of fluorescence light microscopy (FLM) by means of the Meteor system from delmic. This will allow users to perform in situ CLEM experiments in the vacuum of the cryoFIB/SEM and will facilitate the identification and conformation of regions of interest before and during milling. This will greatly increase the chance that the region of interest will be retained in the milled lamellae when imaged in one of the eBIC Krios's; 3) During 2022 the eBIC team integrated the Dectris SINGLA detector, a hybrid-pixel electron-counting camera to assess its capabilities for micro-electron diffraction experiments. The detector was installed on the Research Complex at Harwell Glacios™ 200 kV Cryo-TEM that is hosted and operated in partnership with eBIC. A coordinated effort with the DIALS group, the HeXI instrument, CCP-EM and CCP4 allowed us to establish a rapid and automated workflow for the collection of microED data. An access program is currently in development, and we expect to start with friendly users during the summer of 2023; 4) Data management and automation of data processing for users has been considerably simplified by the development of Murfey both for single particle cryo-EM and Cryo-ET. Murfey automates the data transfer and processing pipelines for both single particle cryo-EM and cryo-ET at eBIC; 5) A new on-the-fly cryo-ET processing pipeline has been implemented, which generates reconstructed 3D volumes allowing the users to assess their data as it is collected, making sure that only the best data is collected and used for further analysis; 6) eBIC is delighted to be a partner in the Horizon Europe project Fragment-Screen (https://fragmentscreen.org/home), with Diamond's participation facilitated by funding from the UKRI Horizon Europe guarantee scheme. The project started on the 1st of February 2023.

B24 has also had another strong year with the beamline now fully commissioned and users able to access both instruments (B24's full field cryo X-ray transmission microscope for soft X-ray tomography (cryoSXT) experiments and the bespoke cryo-Structured illumination Microscope (CryoSIM) for 3D super-resolution fluorescence cryo-imaging). The team are refining correlative workflows with cryoSXT and CryoEM which have been pioneered at B24. A major effort continues in fostering and growing the user community and the last year has been busy with the team raising awareness of the unique capabilities of B24 at both national and international conferences together with focused workshops to train and educate users.

Simplifying the B24 user data collection and processing experience is progressing nicely with the team automating part of the workflow through further development of the B24 software pipeline. Some of the workflow and software developments have been supported through the Horizon 2020 project iNEXT-Discovery (https://inext-discovery.eu/) and successfully delivered for the wider community. The team has also developed additional functionality by providing for tomography experiments to be carried out at the iron and calcium edges.

A reminder that access for European users of both eBIC and B24 continues to be supported through iNEXT-Discovery which runs till the end of July 2024. As an iNEXT-Discovery user, access and support is fully funded and European based groups are encouraged to apply through the iNEXT-Discovery user portal.

Finally, the BCI user meeting continues to grow and in partnership with the CCP-EM spring symposium this year's hybrid meeting surpassed our expectations with 225 participants in person and 484 zoom attendees attending the BCI user meeting which kicked off a very enjoyable 3 days of talks from users and the wider community. We look forward to seeing many of you at next year's meeting which will again be hosted at the East Midlands Conference Centre in Nottingham.

> ***"A major effort continues in fostering and growing the user community and the last year has been busy with the team raising awareness of the unique capabilities of B24 at both national and international conferences together with focused workshops to train and educate users."***

Figure 1: the B24 team.

Figure 2: the eBIC team.

The atomic structure of alpha-synuclein filaments of Parkinson's disease

Neurodegenerative Diseases – Non-Communicable Diseases – Health & Wellbeing – Neurology – Structural Biology – Life Sciences & Biotech

Parkinson's disease is a neurodegenerative disorder that affects millions of people around the world. It is a chronic and progressive disease that primarily affects the motor system, causing a range of symptoms such as tremors, rigidity, and difficulty with movement and balance. Although Parkinson's disease is most commonly diagnosed in older adults, it can also affect younger people.

In many neurodegenerative diseases, one or a few different proteins form aggregates, *i.e.* amyloid filaments, in the brain. In Parkinson's disease, the amyloid filaments are made of the protein alpha-synuclein. For the past six years, it has been possible to determine the 3D atomic structures of amyloid filaments extracted from the brains of people who have died with a neurodegenerative disease, using cryo-Electron Microscopy (cryo-EM). During that time, many structures have been solved, including the structure of amyloid filaments of the protein tau from the brains of individuals with Alzheimer's disease. However, the structures of the filaments from Parkinson's disease remained unknown. One of the difficulties was that many of them do not twist, which leads to technical problems in the structure determination process. In this study, an international team of researchers solved the structure of alpha-synuclein filaments. They collected many electron microscope images at eBIC and sifted through them all to find the minority of the filaments that did twist.

Besides looking at alpha-synuclein filaments from the brains of individuals with Parkinson's disease, they also looked at alpha-synuclein filaments from the brains of individuals with dementia with Lewy bodies (DLB). Although these are two different diseases, they found that alpha-synuclein filaments from the two diseases are identical. They have named this α-synuclein structure the Lewy fold. This observation agrees with similar neuropathological features in these brains, i.e. the presence of Lewy bodies.

The discovery that α-synuclein filaments have identical structures in both diseases indicates that they share a continuum. However, this is not the case for some other neurodegenerative disorders characterised by an abundance of α-synuclein filaments. The research group had previously examined the structures of filaments from multiple system atrophy (MSA) and found them to be vastly different from the Lewy fold.

Knowledge of the structure of the alpha-synuclein filaments can be used to develop new molecules that could be useful in the clinic. For example, it may now be possible to use structure-based design to develop small molecules as new ligands for positron emission tomography (PET) specific for Lewy and MSA folds. Such ligands allow the presymptomatic detection of α-synuclein assemblies in brain tissues, which is crucial to early intervention. The results can also be used to develop better model systems of disease and will help to develop methods for producing α-synuclein filaments with structures identical to those in human brains. In the long term, this work could lead to the development of new therapies for both Parkinson's disease and dementia with Lewy bodies.

Related publication:
Yang, Y. et al. Structures of α-synuclein filaments from human brains with Lewy pathology. *Nature* **610**, 791–795 (2022). DOI: 10.1038/s41586-022-05319-3

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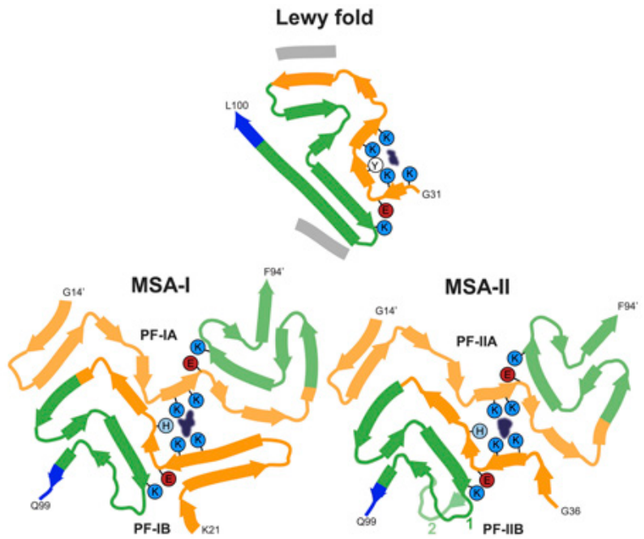

Secondary structure elements in the Lewy and multiple system atrophy (MSA) folds.

Understanding a protein that fuels bowel cancer

Non-Communicable Diseases – Health & Wellbeing – Cancer – Biochemistry – Chemistry – Structural Biology – Biophysics – Drug Discovery – Life Sciences & Biotech

Tankyrase is an important protein that regulates a wide range of processes relevant to cancer and other conditions, such as diabetes, neurodegeneration and fibrosis. It supports 'Wnt signalling', essential for cell division and development and maintaining stem cells. Tankyrase also controls other cell functions critical to cancer, including the maintenance of telomeres at the end of chromosomes. Therefore, tankyrase has received substantial attention as a potential drug target.

Tankyrase is part of the 'PARP family' of proteins, and drugs blocking PARP1 are already in clinical use. However, tankyrase remains poorly understood, with scientists unsure of how the protein is switched on, how it functions and how to block it without causing unwanted side effects.

Tankyrase self-assembles to form filamentous polymers, but how polymerisation contributes to tankyrase function and catalytic activity was unknown.

Scientists at The Institute of Cancer Research in London used cryo-Electron Microscopy (cryo-EM) at eBIC to investigate the architecture of tankyrase filaments. In particular, they were keen to identify any potential contacts made by the catalytic domains, as these interactions may control the effect of polymerisation on tankyrase's activity.

Using helical reconstruction, they revealed the architecture of a tankyrase filament containing the polymerisation and catalytic domains. Surprisingly, the filament turned out to be a double helix, something they didn't anticipate based on previous X-ray crystallography studies. Their results revealed extensive interactions between different domains of tankyrase, including those involving the catalytic domain. Based on subsequent biophysical, biochemical and cell-based studies, the researchers proposed that a polymerisation-induced allosteric switch regulates tankyrase's catalytic functions.

The scientists draw parallels between the activation mechanism of PARP1 and tankyrase for the first time. Similarly to PARP1, they suggest tankyrase works by being recruited to a specific site and 'self-assembling', activating itself by clustering and changing its 3D structure.

Although previous research has developed drugs that block tankyrase - in the hope of treating bowel cancer - they caused too many side effects to reach clinical trials. That's likely due to tankyrase being involved in such a wide range of processes, or essential functions of Wnt signalling in normal cells. This work provides novel insights into fundamental biological mechanisms but should also enable the development of novel tankyrase inhibitors and overcome the limitations of currently available molecules.

Related publication:
Pillay, N. et al. Structural basis of tankyrase activation by polymerisation. *Nature* **612**, 162–169 (2022). DOI: 10.1038/s41586-022-05449-8

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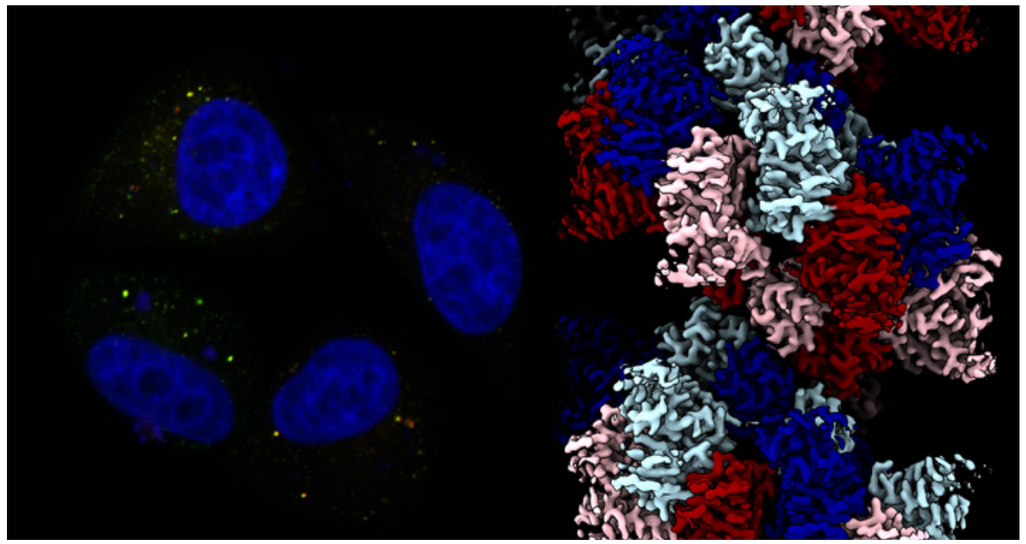

The signalling protein tankyrase forms punctate structures in cells (left, tankyrase shown in green with nucleus in blue). These puncta appear as tankyrase self-assembles into chain-like fibres. Cryo-electron microscopy of the isolated tankyrase protein enabled researchers to decipher the molecular structure of these fibres (right) and learn how tankyrase is activated by self-assembly.

Understanding the Power of Movement in Plants

Plant Science – Biochemistry – Agriculture & Fisheries – Chemistry – Structural Biology – Biophysics – Life Sciences & Biotech

Auxins are hormones playing a central role and controlling nearly all aspects of plant growth and development. Charles Darwin observed that plants could grow directionally in response to environmental stimuli such as light or gravity. In his book, *The Power of Movement in Plants***, published in 1880, Darwin showed that the part of the plant responding to such a stimulus differs from the part that perceives it. He proposed that some kind of 'influence' must travel from the perception site to the response area. However, Darwin was unable to identify the influence.**

Darwin's 'growth accelerating substance' was identified in 1926 as the hormone auxin. Later research identified that auxin is the growth factor that determines almost all plant responses to environmental changes. Directional transport of the auxin molecule between cells is required to ensure that the auxin response occurs in the correct part of the plant.

It wasn't until the 1990s that scientists identified the proteins involved in the process. PIN-FORMED (PIN) proteins are auxin transporters, and they are essential for the development of auxin gradients within plant tissues that guide plant growth. They're named from the distinct needle-like 'pin' form, without shoots or flowers, into which plants with dysfunctional PIN proteins grow. Even then, how PIN proteins fold, how they recognise substrates and inhibitors and the molecular mechanism behind transport have remained unknown.

Now researchers from Aarhus University and the Technical University of Munich have used single particle cryo-EM at eBIC to provide the first structural basis of auxin transport by PIN proteins. By combining three structures of *Arabidopsis thaliana* PIN8 with a comprehensive biochemical characterisation, their results finally provide the molecular mechanism behind auxin transport. They reveal an elevator-type transport mechanism similar to sodium/proton antiporters, bile acid/sodium symporters and bicarbonate/sodium symporters.

Their work provides a comprehensive molecular model for auxin recognition and transport by PINs and links and expands on a well-known conceptual framework for transport. In addition, it explains a central mechanism of polar auxin transport, a core feature of plant physiology, growth and development. Furthermore, their results offer insights into how a broad range of widely used herbicides, collectively known as synthetic auxins and anti-auxins, can be recognised by PIN proteins. It could therefore help to create more specific herbicides and minimise their environmental impact.

Related publication:
Ung, KL. et al. Structures and mechanism of the plant PIN-FORMED auxin transporter. *Nature* **609**, 605–610 (2022). DOI: 10.1038/s41586-022-04883-y

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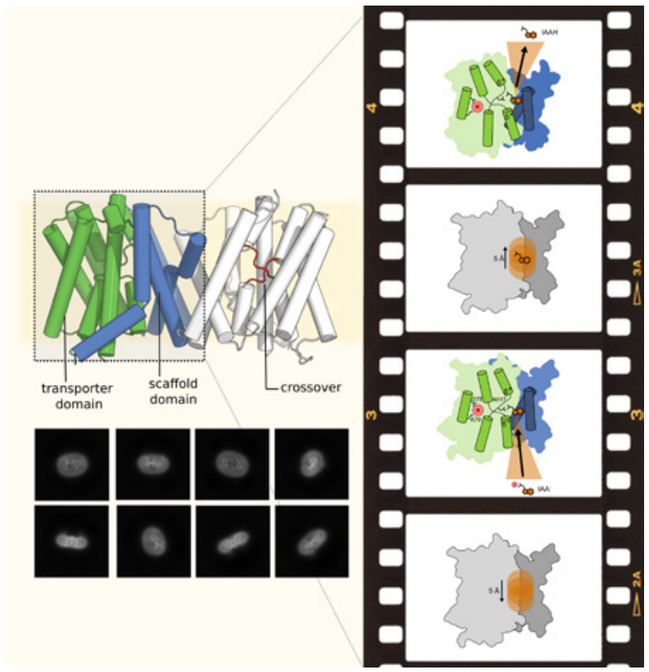

PIN8 is a 40 kDa membrane protein that transports the plant hormone Auxin. It forms a homodimer with each monomer containing two domains: transporter (green) and scaffold (blue). In the transporter domain a distinct crossover (red) is localized at the middle of the membrane plane that defines the auxin binding site. Below the structure show 8 representative 2D classes from the data collected at eBIC that resulted in 3 distinct conformations solved. To the left a schematic of the transport of Auxin (IAA) with two key conformations coloured that summarizes the transport mechanism as described by the data obtained at eBIC is shown.

Deciphering a methane-oxidising enzyme using cryo-Electron Tomography

Earth Science & Environment – Biotechnology – Climate Change – Biochemistry – Chemistry – Structural Biology – Engineering & Technology – Life Sciences & Biotech

Methane is a greenhouse gas whose atmospheric concentration is currently around two-and-a-half times greater than pre-industrial levels and is increasing steadily. This rise has important implications for climate change. Methane-oxidising bacteria (methanotrophs) play a central role in greenhouse gas mitigation and have potential applications in biomanufacturing. Their primary metabolic enzyme, particulate methane monooxygenase (pMMO), is housed in copper-induced intracytoplasmic membranes (ICMs). pMMO's methane oxidation activity critically depends on its lipid environment, as detergent-solubilised enzymes used for crystallization or single particle cryo-EM structural analysis show no enzymatic activity. Therefore, to fully understand the molecular mechanism of pMMO, it is essential to study it in its native membrane environment.

A team of scientists used serial cryo-Focused Ion Beam (cryoFIB) milling/Scanning Electron Microscope (SEM) volume imaging and lamellae-based cellular cryo-Electron Tomography (cryo-ET) to study pMMO in native cells. They showed that these pMMO-embedded ICMs are derived from the inner cell membrane. Furthermore, pMMO forms ordered hexagonal arrays of trimers in intact cells. The structure of pMMO trimer in the ICM, composed of PmoA, PmoB and PmoC (88 KD together), was resolved by cryo-ET and subtomogram averaging to 4.8 Å resolution. Data were collected at Diamond's electron Bio-Imaging Centre (eBIC) using the 300 kv Titan Krios microscope with technical support from staff scientists at eBIC. The structure reveals that the critical helices, which were missing or disordered from previous structural studies, are stabilised by the lipid membrane and form an active Cu centre for enzyme function. pMMO array formation correlates with increased enzymatic activity, highlighting the importance of studying the enzyme in its native environment.

The remarkable hexagonal arrays of pMMO trimers in the native ICM provide new insights into the mechanistic understanding of the stimulated enzymatic activity of pMMO in intact cells. Understanding how pMMO arrays assemble and promote methane oxidation will be integral to future efforts to deploy methanotrophs in biotechnology. The findings also demonstrate, for the first time, the power of cryo-ET to structurally characterise small (< 100 KD) native transmembrane enzymes in the cellular context.

Related publication:
Zhu, Y. et al. Structure and activity of particulate methane monooxygenase arrays in methanotrophs. *Nature Communications* **13**, 5221 (2022). DOI: 10.1038/s41467-022-32752-9

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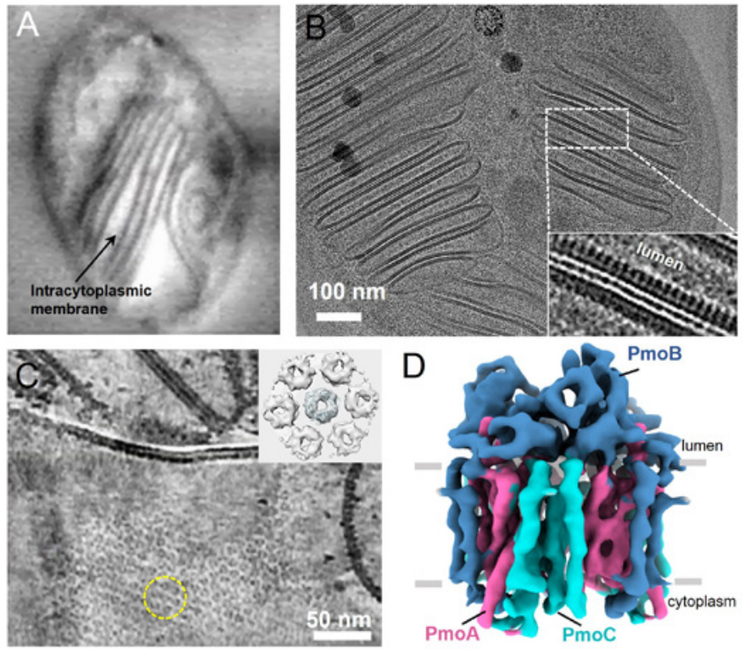

Native pMMO structure from methanotrophic bacteria. (A) A slice from serial cryoFIB/SEM volume of M. capsulatus (Bath). (B) A projection image of cryoFIB lamella of Bath. Inset, enlarged view of boxed area, displaying comb-like edge-on view of membrane-bound protein arrays. (C) A tomographic slice of Bath lamella displaying hexagonal arrays of particles. Inset, a subtomogram average of the 7-particle volume (yellow circle). (D) The structure pMMO trimer in ICM at 4.8 Å resolution, with each pMMO monomer consisting of PmoA (pink), PmoB (blue), and PmoC (cyan).

Herpes simplex virus makes itself at home by rearranging the contents of your cells

Technique Development – Life Sciences & Biotech - Pathogens – Health & Wellbeing – Structural biology

Herpes simplex virus (HSV)-1 is a highly prevalent human pathogen that causes life-long infections that usually manifest as cold sores or genital herpes. However, HSV-1 infection can cause life-threatening disease (encephalitis), and people with weakened immune systems such as neonates, the elderly and patients on certain immunosuppressive drugs tend to be more at risk. HSV-1 has also been developed as a potential therapeutic; it is already in use as an anti-cancer agent (T-VEC) and is under development as a delivery vector for gene therapy. Given both the biotechnology potential of engineered HSV-1 and the detrimental health effects of infection with the wild virus, it is essential that we understand more about how this virus interacts with infected human cells.

Infection of human cells with HSV-1 has been extensively studied using imaging techniques like electron and fluorescence microscopy, but these techniques can generally only image thin sections of the cell or work at low resolution. Additionally, many of these techniques rely upon sample processing that can distort the shape of the cell and its components. Researchers from the University of Cambridge sought to image infected cells that are as close as possible to how they would look in the cellular context. Generally, before imaging cells undergo a series of steps to aid in their imaging and can lead to changes in cell structure that lead to artefacts and generate images that are not representative of their cellular state. Elimination of these states and flash-cooling of cells to liquid nitrogen temperatures preserves the cells structures and provides a way to image cells in a 'near-native' state. This opens up the possibility to understand, in this instance, how the shape and arrangement of cellular compartments change through the course of virus infection.

They used the facilities on Diamond's B24 beamline to flash-freeze infected cells in liquid ethane, cryogenically preserving them in a 'near-native' state, and to perform cryo-soft-X-ray tomography (cryo-SXT). The cryo-SXT analysis allowed them to reconstruct 3D images of infected cells at very high resolution. Combining this analysis with a special fluorescent 'timestamp' strain of HSV-1, constructed in Cambridge, allowed them to work out the stage of infection each cell was in when it was frozen.

Using B24, they were able to identify individual virus particles within the infected cells and to see how virus infection progressively changes the shape and distribution of mitochondria (the powerhouses of the cell), vesicles (the cell's internal delivery system) and lipid droplets (both energy stores and signalling stations for the immune system). Importantly, the high throughput imaging afforded by B24 allowed them to define these changes numerically.

This study demonstrates the power of cryo-SXT for monitoring virus infection and highlights which organelles to focus on as we study the molecular characteristics of herpesvirus infection.

Related publication:
Nahas, KL. et al. Near-native state imaging by cryo-soft-X-ray tomography reveals remodelling of multiple cellular organelles during HSV-1 infection. PLoS Pathogens **18**, 7: e1010629 (2022). DOI: 10.1371/journal.ppat.1010629

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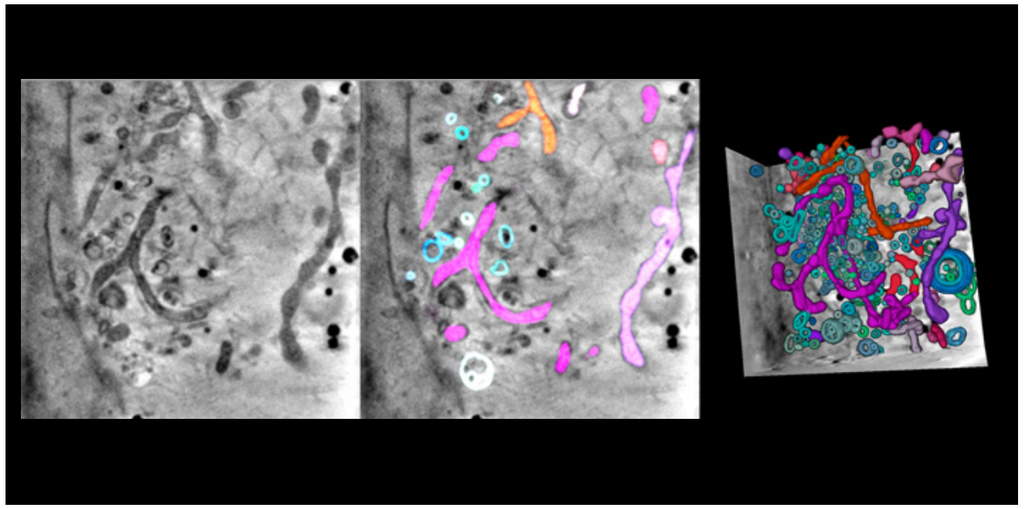


Herpes simplex virus infected cell as visualised using cryo-soft-X-ray tomography (left) allows identification of cellular structures (middle) that can be visualised in 3D (right), showing vesicles (rings) and mitochondria (tubes) in the infected cell.

Structures and Surfaces Group

Chris Nicklin, Science Group Leader

This year has been another exceptionally busy year for the Structures and Surfaces group, with the number of on-site user visits returning to pre-pandemic levels, whilst the experiments become ever more complex. *In-situ* and *operando* studies have been a real focus for the group, whether it is straining a sample for electronic structure measurements, employing 'realistic' conditions to study catalytic processes or understanding the processes occurring under electrochemical control amongst many others. Many of these developments have been undertaken collaboratively with key groups, but with the resulting environments being made available to all users. Increasingly, developments are being undertaken across the group and with other groups to ensure consistent approaches that will benefit the user community; for example, we have just started a new ultrahigh vacuum manipulator design that will integrate sample biasing and the potential for strain control across four beamlines at Diamond. The Structures and Surfaces Group includes four beamlines: I05 (Angle Resolved Photoelectron Spectroscopy – ARPES), I07 (Surface and Interface X-ray Diffraction), B07 (Versatile Soft X-ray Scattering – VERSOX), and I09 (Atomic and Electronic Structure of Surfaces and Interfaces). They offer a variety of techniques to examine the atomic scale structure, chemical nature and electronic state at buried interfaces or the surfaces of materials. The group has continued to develop its strategy, outlining the facilities that we plan to offer as part of the Diamond-II programme. The important role that surfaces and interfaces play in broader research areas such as battery technology, photovoltaic structures, the discovery of novel quantum materials and catalytic/electrochemical systems under *operando* conditions are key drivers for these developments. Below we highlight some of the important developments on each instrument over the last year.

Beamline I05:

This world leading beamline delivered a suite of major upgrades including a new analyser on the high resolution end station, innovative capillary optics on the nanoARPES branch and a new high-flux grating to ensure that the ARPES experiments give as clear a view as possible of the sharp electronic bands in novel materials. The implementation of the 'deflector mode' allows angle resolved measurements to be recorded without having to physically move the sample, increasing the speed of data acquisition and maintaining the photon beam in a consistent position on the sample. For nanoARPES, excellent data quality is achieved through the ability to focus the beam onto high quality areas of the sample, which in many newly produced crystals may only be a few micrometres in size. Understanding how sample heterogeneity influences the electronic structure remains a very active focus of research on the beamline. The team on I05 is leading the development of a new manipulator design to integrate electrical contacts that will enable direct biasing of the sample or control of a piezoelectric device to apply mechanical strain to a crystal. Strain device development is the subject of a joint appointment with the group of Clifford Hicks at the University of Birmingham, with miniaturisation of the device a key goal to enable rollout of these studies across multiple beamlines at Diamond. The scientific programme on the beamline continues to focus on novel new materials, including 2D materials and heterostructures, superconductors, magnetic systems and topologically non-trivial materials with some of the properties studied including charge density waves and the role of strain in adapting the electronic structure.

Beamline I07:

The team on the surface and interface diffraction beamline I07 has continued to focus on a number of key upgrades to enhance the capabilities. Continuous (fast) scanning of the diffractometer is now implemented for both experimental end-stations such that multi-axis motions enable scans along a specific trajectory in reciprocal space. Automated fast attenuators have been installed and integration with the detector and data acquisition software is currently being implemented. This will enable fast measurement of crystal truncation rods that cover many orders of magnitude changes in intensity. The Large Area Detector positioning system has been upgraded to extend the range of sample-detector distances that can be achieved, improving the grazing incidence small (wide) angle X-ray scattering (GIS(W)AXS) investigations. In addition, new implementations of Soller slits have been integrated to improve the signal to noise levels for several types of experiment. A long term ambitious plan continues to be discussed, to upgrade the optical layout of the beamline, switching from mirror focusing to making use of compound refractive lenses to provide additional tunability in the beam dimensions as well as a smaller ultimate spot size. A concurrent upgrade to the double crystal deflector (DCD) system would improve the ease of operation and enhance data quality for diffraction studies from liquid surfaces. The I07 team is also moving forward with improving the analysis software, in particular for visualisation of reciprocal space maps and reduction/extraction of crystal truncation rod data. The focus of research on the beamline continues to expand with new types of experiments implemented in the fields of electrochemistry, perovskite solar cells and molecular monolayers.

Beamline B07:

The second branch (B07B) of the VERSOX beamline expanded its number of user experiments in 2022, with the second end-station for UHV high-throughput X-ray photoelectron spectroscopy (XPS) studies, to enable chemical state analysis of many samples, having its first users. The initial end-station, developed in collaboration with Sven Schroeder's group from the University of Leeds, for near edge X-ray absorption fine structure (NEXAFS) studies using soft X-rays to study materials such as organic molecules, battery materials, and catalysts has proven to be exceptionally productive. This branch has continued to improve through the integration of a vortex detector and very recently a UV-Vis spectrometer to correlate with the NEXAFS studies. The near ambient pressure XPS (NAP-XPS) system available on Branch C of the beamline continues to work well and has been enhanced by the implementation of a channel cut crystal monochromator that increases the flux at higher energies whilst also improving the energy resolution. Many of the studies on B07 focus on catalytic systems, electro- and photochemistry under *operando* conditions. The automated gas-rig and the close proximity of a state-of-the-art nanoparticle source (in collaboration with Richard Palmer from the University of Swansea) have added significant capabilities to the research portfolio at the beamline.

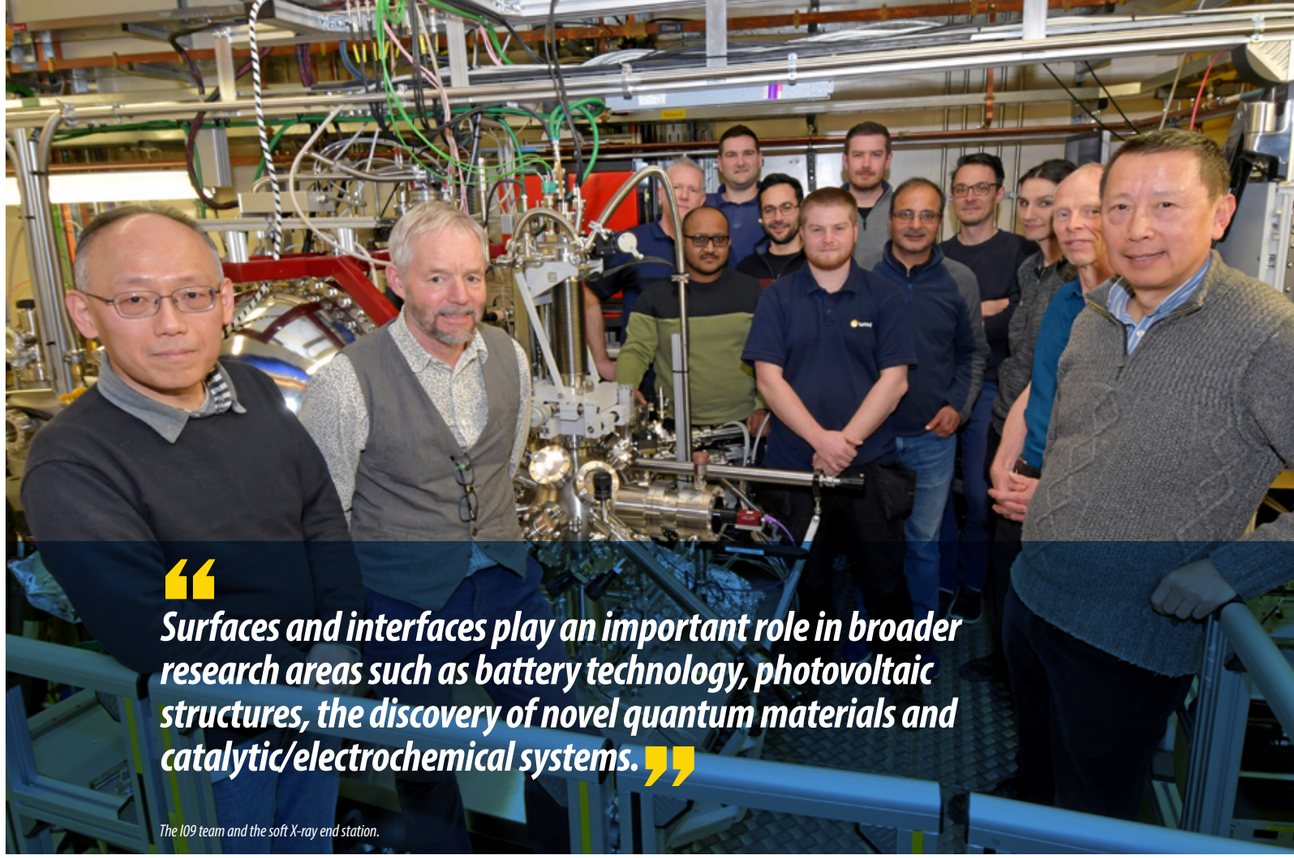
Beamline I09:

Beamline I09 has maintained a very active user programme whilst also working on a number of significant developments. The end station for the soft X-ray ARPES system is now complete and the momentum microscope analyser has started to be commissioned. This is a novel design with the first k space images of dispersion from gold being realised in March 2023. Extensive commissioning is required to optimise the voltages on all of the (more than 40) lens elements to make the system available for user operation. This instrument will complement the ARPES facilities available at I05, working at higher energies and improving the ability to probe buried or true bulk electronic states and their k_z dependence. I09 also took delivery of a small spot ultraviolet photoelectron spectroscopy (UPS) system that is essential to develop the momentum microscope to reach its ultimate resolution without having to use the synchrotron beam. Concurrently, the software to drive the analyser is being developed with the detector manufacturer, to ensure that it is maintainable and can be driven by the Diamond control system. Smaller projects, focusing on delivering a more robust sample manipulator are ongoing with assembly and commissioning underway. A wide research programme is supported by I09, ranging from surface science experiments on molecular adsorption or single atom catalysts through to conventional and solid state battery studies.

The group are enhancing the associated infrastructure available, including the design of a new offline ultrahigh vacuum system to characterise samples, a proposal that was well received by the Scientific Advisory Committee (SAC). Design work has started on a small part of this system that will initially be operated as a basic standalone chamber. This continues to be a high priority that will position Diamond to be able to rapidly study new samples and enhance the link between laboratory and synchrotron based experiments. We plan for this capability to be at the core of many of the joint PhD studentships that we support.

The variety of science undertaken within the group is very large, underpinning fundamental surface science investigations through to understanding the relevance to real-world applications. The user highlights include how I05 has been used to study 'misfit compounds' formed from stacked two-dimensional materials with no epitaxial relationships between the layers. The influence on the electron bands caused by the misfit can be probed using the small spot of nanoARPES branch. A different type of disorder is highlighted in the work from I09, where the structure of γ-Ga_2O_3 has been studied. This material has many potential uses in sensors, solar cells and electronics but can exist in a large number of phases that must be understood to fully exploit and engineer them. On I07 researchers probed how sulfobetaines are incorporated into a lipid bilayer that mimics a cell membrane finding that the roughness increases together with other significant structural changes at higher concentrations. The role of these molecules as potential drug-delivery agents is outlined in this research. The work on B07 (Branch C) has utilised the tunability of the photon source to use resonant XPS to study ionic liquids and how they perform when a voltage is applied to probe their use in electrochemical storage devices. The ability to probe a particular element (*e.g.* cobalt) has enabled real detail to be added to the understanding in these systems. The highlight for B07 (Branch B) has focused on the other main technique available at the beamline, NEXAFS, which was used to understand co-crystallisation in a two-component system to understand how such materials could be used in future drug delivery processes that could tailor drug-release profiles.

The members of the Structures and Surfaces group are committed to continue offering the best support to our users, to ensure the highest quality scientific output from the beamlines. The combination of strong interactions and collaborations, together with continuous improvements to the instrumentation, software and technique development is key to our success. Please contact us if you would like to discuss any of the possibilities that we offer and how such synchrotron based studies could help in your research.



The I09 team and the soft X-ray end station.

More than the sum of its parts: The electronic properties in a stack of crystals

Physics – Hard Condensed Matter – Structures – Materials Science

Traditional solid state physics focuses on crystals with a well-defined periodicity. However, in recent years it has become increasingly clear that stacks of thin crystals with non-compatible periodicities can bring about new properties that are different from those of the constituent parent crystals. Researchers from Aarhus University in Denmark wanted to develop a better understanding of this phenomenon.

Most stacks of crystals with non-compatible periodicities are artificially made by bringing two-dimensional materials into contact with each other. Rather than using such an artificial crystal, the research team chose to investigate a so-called misfit compound. A misfit compound is a naturally occurring infinite stack of two-dimensional materials with incompatible periodicities and hence an easily accessible material to test the effect of stacking layers in incompatible periodicities. Their chosen misfit compound was a stack of square and hexagonal layers with no common periodicity.

Using Diamond's I05 nanoARPES branch allowed them to study the surface with a very high spatial resolution. This is necessary because the misfit crystal is a stack of two types of layers. It can have two terminations at the surface with either one or the other layer. The areas of these different terminations are quite small, so the beam spot used for probing must be strongly focused to probe only one of them. As the angle-resolved photoemission technique they used is very surface-sensitive, the underlying layers do not contribute strongly to the measured signal. The measurement is thus dominated by the surface layer.

Their results showed that the properties of each layer strongly resemble those expected for a free-standing version of that layer, without the influence of the other layer. However, there were some new properties arising in the form of one-dimensional electronic states.

The findings provide a new way to create one-dimensional electronic states. Such states have interesting fundamental properties that could potentially be used for next-generation electronic devices in the future.

Related publication:
Chikina, A. *et al.* One-dimensional electronic states in a natural misfit structure. *Physical Review Materials* **6**,(2022). DOI: 10.1103/PhysRevMaterials.6.L092001

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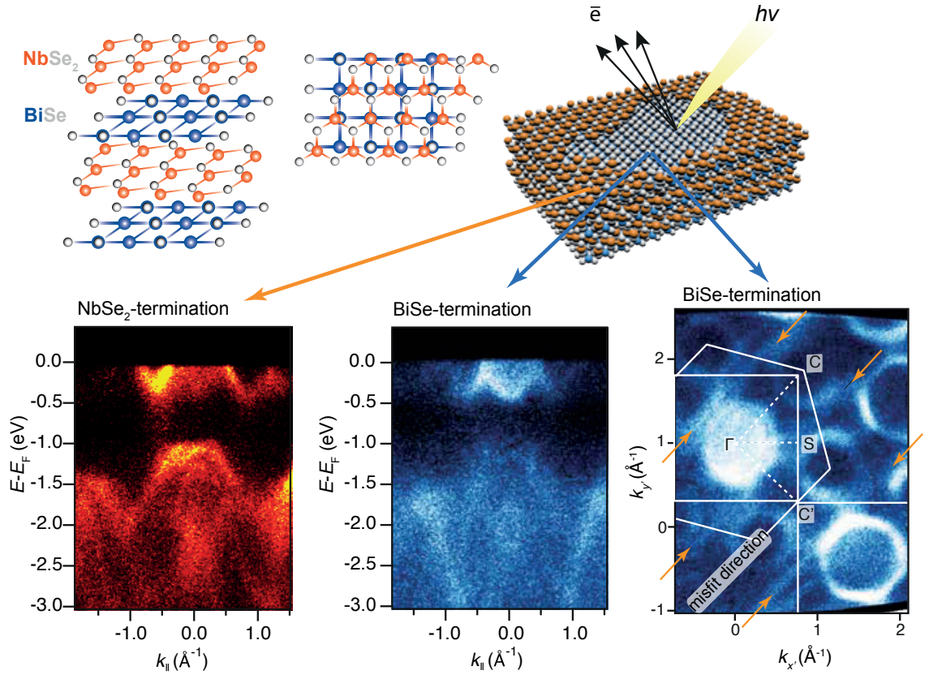

Crystal structure of the misfit compound used in this study. The compound is a stack of square and hexagonal layers and the lattice constants of the two layers do not quite fit in length. A surface of the misfit crystal shows areas terminated by one or the other type of layer and the beam from Diamond is sufficiently focused to probe just one area of termination. The bottom row shows experimental data from the two terminations in different colours. In the image on the right hand side, line-like features appear that are not expected for any of the constituent layers. They emerge due to the interaction of the two materials.

Mixing it up: can sulfobetaines help with drug delivery?

Surfaces – Drug Delivery – Physics – Physical Chemistry – Health & Wellbeing – Chemistry – Interfaces and thin surfaces – Life Sciences & Biotech

The cell membranes of almost all organisms are formed of two layers of lipid molecules that prevent ions, proteins and other molecules from entering or leaving the cell. Vesicles are small, membrane-bound structures used to transport substances within a cell, and scientists can engineer vesicles to carry drugs to where they are needed. Engineered vesicles typically use lipids to form a membrane around the drug molecules.

As phosphocholine (PC) phospholipids are the main component of natural lipid bilayers, they are often used as simple models to study whether drug compounds are likely to be able to enter the cell. Phospholipids are non-toxic to cells and potentially useful for drug delivery and other medical applications. However, they are neither cheap nor easy to synthesise, unlike sulfobetaine (SB) based lipids. SB lipids are non-toxic and used in medical applications such as commercial eye drops, but their structure is less well studied.

Researchers from the University of Bath conducted experiments to investigate the structure of mixed monolayers of sulfobetaines and phosphocholine phospholipids. Using a monolayer simplifies the system and makes it 2D rather than 3D. They wanted to explore the interactions between the two types of lipids, which have opposite charge distributions in their head groups, and how they affect the properties of the mixed monolayers. They created lipid mixtures with different ratios of sulfobetaines and phospholipids and used a combination of X-ray Reflectometry (XRR) studies on Diamond's I07 beamline and Neutron Reflectometry (NR) experiments at ISIS Neutron and Muon Source to investigate the structure of the mixed monolayers. During the experiments, the research team also manipulated the surface pressure, developing an understanding of the nature of the structure and how it changes with pressure.

The team found that adding the sulfobetaine to the mixture did not significantly affect the monolayer structure. However, the SB tails are longer than the PC tails, making the layer surface appear rougher. At higher SB concentrations, the data showed the phospholipids behaved normally, but that the sulfobetaine molecules were closer to perpendicular to the water surface. This suggests that the two molecules have different configurations at these higher concentrations, making successful preparation of vesicles unlikely, as they won't pack well together. A very high concentration of sulfobetaines would probably also interact badly with cells, making the mixture toxic.

Their results confirm that the interactions between sulfobetaines and phospholipids are favourable and that these combinations have potential applications in future drug delivery methods. With their opposite charge distribution, sulfobetaines may also interact differently with drug components, facilitating the delivery of drugs that wouldn't be possible with phospholipids alone.

Related publication:
Elstone N *et al*. Structural investigation of sulfobetaines and phospholipid monolayers at the air-water interface. *Physical Chemistry Chemical Physics* **24**, 22679-22690 (2022) DOI:10.1039/D2CP02695C

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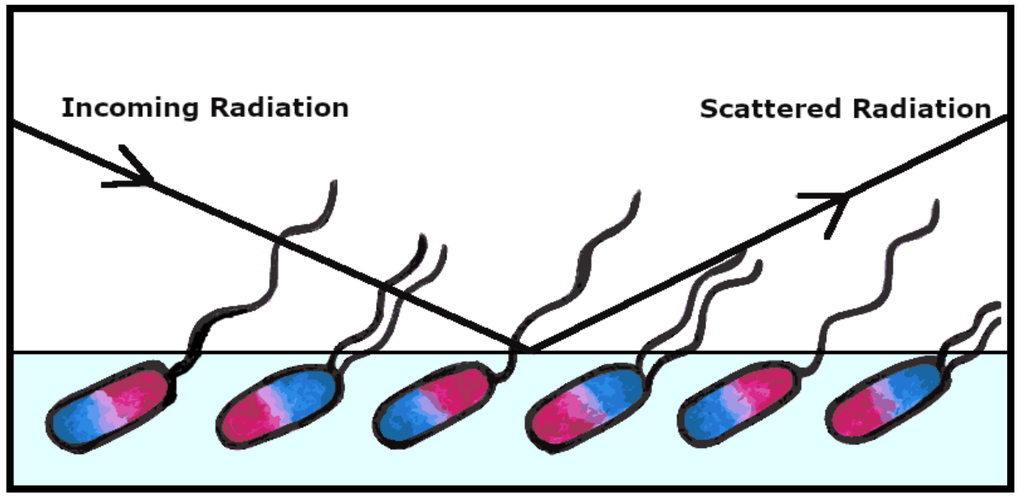

Artistic representation of the possible structure of the 1 : 1 DMPC : SB3-18 mixtures monolayer at the air–water interface from the results obtained from fitting NR and XRR data.

New NEXAFS beamline rapidly characterises bonds in non-crystalline materials

Physical Chemistry – Technique development – Chemistry

Drugs are commonly manufactured in the form of tablets that contain other materials. The role of these additional ingredients is to ensure that drug release in the body is controlled, with just the right dose over a defined amount of time. There is a lot of interest in the idea of manufacturing the drug itself by crystallisation with a second ingredient, giving access to drug release profiles that cannot be achieved through tablet formulation.

The products of crystallisation with a second component are typically held together by electrostatic bonds associated with the sharing of hydrogen atoms between the two components. Such bonds are called hydrogen bonds. Often, the hydrogen nucleus separates from its electron and forms an ionic bond, and the resulting product is called a salt. Clarity about which type of bonding occurs is essential for predictive modelling of properties (*e.g.* for designing a drug release profile), as well as for regulatory approval and patenting of the resulting medicine.

Traditionally, crystal structure analysis by X-ray diffraction (crystallography) has been used to classify products into co-crystals and salts. Crystallography relies on locating the position of the hydrogen in the crystal structure precisely, which can be a challenge because hydrogen is an extremely weak X-ray scatterer.

Diamond and the University of Leeds partnered to develop the new high-throughput Near-Edge X-ray Absorption Fine-Structure (NEXAFS) spectroscopy end station at the B07 beamline. The team then used it to characterise three 2-component systems to examine whether the sensitivity of NEXAFS to the hydrogen position is sufficient to identify the nature of the intermolecular bond.

The NEXAFS spectra classified the hydrogen donor-acceptor products unequivocally into salts and co-crystals, and even identified that some of the interactions placed the materials on the boundary between salts and co-crystals. As NEXAFS does not require a crystalline form of a sample, this study opens up NEXAFS to characterising hydrogen donor-acceptor interactions in other forms of matter: solutions, melts, thin films, and amorphous materials.

The high-throughput NEXAFS capability of the new B07 beamline facilitates the characterisation of local bonding in organic crystal structures on timescales of minutes. This technique is relevant for a wide range of materials beyond the pharmaceutical context of this work; solar energy conversion, energy storage in batteries, fuel cell power and formulated consumer products (*e.g.* foods and household products such as cleaners) involve hydrogen bonding and proton transfer in non-crystalline matter. The capability to identify these interactions quickly and correctly will be an invaluable aid in the development of new technologies and products.

Related publication:
Edwards, PT. *et al.* Determination of H-atom positions in organic crystal structures by NEXAFS combined with density functional theory: a study of two-component systems containing isonicotinamide. *The Journal of Physical Chemistry A* **126**, 19 (2022): 2889-2898. DOI: 10.1021/acs.jpca.2c00439

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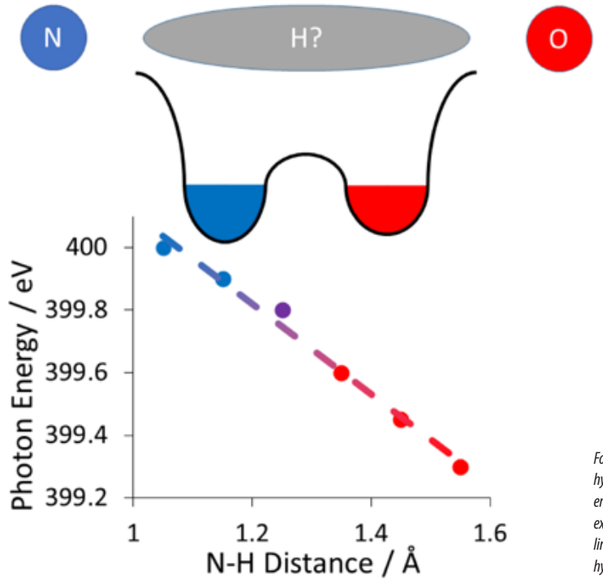

For nitrogen atoms involved in a hydrogen bond, the X-ray photon energy required for a resonant excitation of its 1s electron decreased linearly with the distance to the hydrogen atom.

Using resonant X-ray Photoelectron Spectroscopy to understand the reactivity of ionic liquids

Physical Chemistry – Technique Development - Chemistry

In numerous applications that involve electron donation in a liquid environment, such as electrochemical energy storage and catalysis, a comprehensive understanding of the occupied electronic states is essential to understand and predict reactivity. However, although many theories exist relating electronic structure to reactivity, they have often not been rigorously tested against experimental data. This is especially challenging for solutes in liquids, because the electronic structure of the liquid phase is very hard to measure.

An international team of researchers planned to investigate the electronic structure of both solvents and solutes in solvents. They wanted to explore the limits of the synchrotron technique resonant X-ray Photoelectron Spectroscopy (resonant XPS), which they believe is underused, especially when applied to solvents and solutes dissolved in solvents. These investigations represent a continuation of previous work by the group on Diamond's B07 beamline (Seymour *et al.*, Phys. Chem. Chem. Phys., 2021, 23, 20957), and were carried out on B07-C, the Ambient Pressure (AP) XPS end station.

B07-C is ideal for studying ionic liquid samples. Ionic liquids are made up solely of ions and have very low volatility, which means they can be studied as liquid drops on B07-C XPS. Resonant XPS requires tuneable X-ray energies, which cannot be accessed on lab XPS apparatus.

Simple calculations often capture the electronic structure of the solvents and solutes in solvents, but are not always accurate. For example, the team found they needed more complex calculations to satisfactorily model the cobalt-containing sample studied on B07. The mismatch is caused by unpaired electrons in the cobalt complex; these unpaired electrons give rise to very useful reactivity, but also provide challenges for theory.

Ionic liquids (ILs) are liquid salts made only of positively and negatively charged ions and have a unique combination of properties. ILs are liquid at room temperature, whereas NaCl (common table salt) has a melting point of approximately 1000 K. ILs conduct electricity and decompose before they boil, unlike molecular liquids. These incredible properties mean that there is enormous potential for the use of ILs in electrochemical energy storage devices, *e.g.* batteries for phones and supercapacitors for cars.

For electrochemical energy storage devices, determining what happens when ILs are subjected to potential (*i.e.* electrical energy) is paramount. When the voltage is outside the safe operating range, the IL will decompose, and the device will malfunction. Therefore, the ILs have a voltage operating range, which is analogous to the operating temperature range of people - at high and low temperatures, people malfunction. The factors that control the operating range are very complicated. The results are very useful in understanding how to extend the operating range of ILs used in applications, by linking the electronic data to theory and calculations.

The team has since recorded further results on B07 and is working on testing which theories capture the experimental results recorded.

Related publication:
Seymour, JM. *et al.* Resonant X-ray photoelectron spectroscopy: identification of atomic contributions to valence states. *Faraday Discussions* **236**, 389-411 (2022) DOI: 10.1039/D1FD00117E

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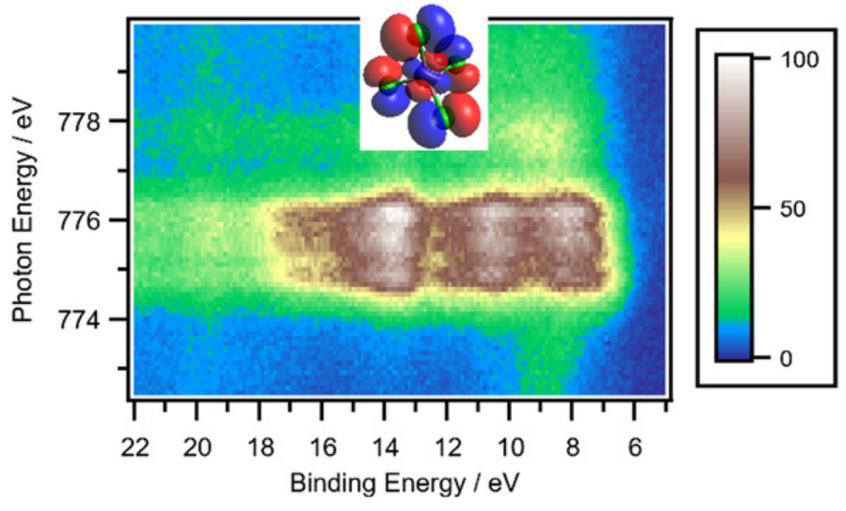

Co 2p$_{3/2}$ resonant X-ray Photoelectron Spectroscopy heat map for the tetrachlorocobaltate anion $[CoCl_4]^{2-}$. The brown/white features capture the energy of the reactive electrons located near to the cobalt atom in the $[CoCl_4]^{2-}$ anion, with a pictorial representation of a molecular orbital depicting calculated electron location also shown.

Taming disorderly oxides

Surfaces – Technique Development – Materials Science – Artificial Intelligence – Physics – Hard Condensed Matter – Structures – Electronics – Energy Materials – Information & Communication Technologies

Gallium oxide (Ga_2O_3) is an interesting electronic material with potential applications in, for example, power electronics, solar-blind UV photodetectors, gas-sensing devices, and solar cells. It exists in a number of different crystal structures (polymorphs), known as alpha, beta, gamma, delta and epsilon.

An international team of researchers wanted to understand how structure influences the electronic structure of one particular phase, γ-Ga_2O_3, which is highly disordered and, therefore, incredibly challenging for experiment and theory. This work was an international collaboration across disciplines, including experimentalists and theorists, chemists and physicists, and synthesis and characterisation experts and combined a machine-learning theoretical approach with experimental results.

They used both soft and hard X-ray Photoelectron Spectroscopy as well as X-ray Absorption Spectroscopy at beamline I09 at Diamond to investigate the electronic properties of γ-Ga_2O_3 and how this relates to its crystal structure. They combined their results with first principles calculations and machine learning (ML). In addition, they used a range of complementary characterisation techniques, including X-ray diffraction, transmission electron microscopy, spectroscopic ellipsometry and photoluminescence excitation spectroscopy.

Using the theoretical approach (including screening one million structures and more than one thousand individual density functional theory (DFT) calculations), they were able to identify a small number of possible structures γ-Ga_2O_3 could realistically have. They then validated this by directly comparing theory with the photoelectron spectroscopy results. By using this combined approach, they could identify good descriptions of both structure and electronic structure of this complex material.

Disordered systems are increasingly interesting for electronic and optical applications. Unlocking their full potential will involve engineering their structure through targeted synthesis in a way that enables finetuning of their electronic and optical behaviour and performance in a device. This is only possible if we have the fundamental knowledge about the relationships between these aspects and the tools to describe and probe them.

Photoelectron spectroscopy is crucial to the exploration of the chemistry and electronic structure of condensed matter, and Diamond houses a number of advanced beamlines that allow the scientific community to use this technique. Hard X-ray Photoelectron Spectroscopy at beamline I09 is particularly crucial as it probes deeper into a material, studying the bulk and buried layers and interfaces, which is important in understanding materials in device applications. This work presents a leap forward in the treatment of complex, disordered oxides and is a crucial step toward exploring how their electronic structure can be understood in terms of local coordination and overall structure. In addition, it showcases a set of tools that the research team successfully applied to a highly challenging material.

Related publication:
Ratcliff, L. E. *et al.* Tackling disorder in γ-Ga_2O_3. *Advanced Materials* **34**, (2022). DOI: 10.1002/adma.202204217

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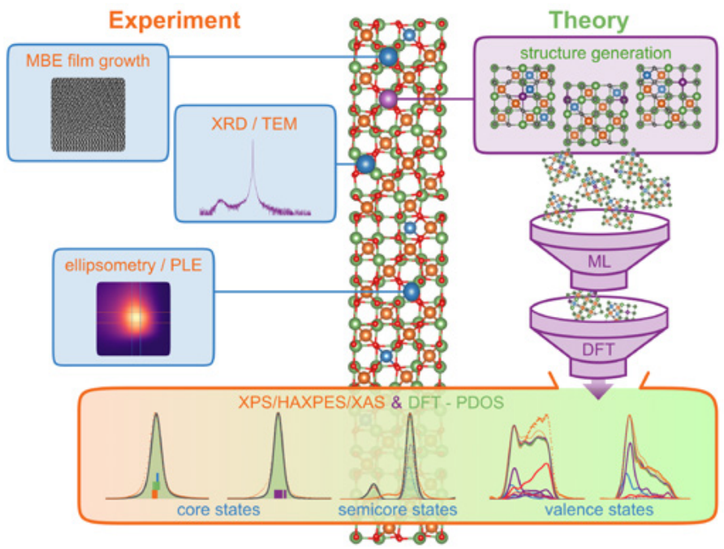


Schematic overview of the parallel experimental and theoretical methods applied to γ-Ga_2O_3. The bottom box displays both experimental X-ray photoelectron spectra as well as theoretical spectra and density of states for core, semicore and valence states. MBE: Molecular-beam epitaxy.

Magnetic Materials Group

Sarnjeet Dhesi, Science Group Leader

The Magnetic Materials Group (MMG) develops and operates a suite of polarised X-ray beamlines to understand new and novel material properties. The beamlines are home to a variety of extremely sensitive material probes including Resonant Elastic/Inelastic X-ray Scattering (REXS/RIXS), X-ray Magnetic Circular/Linear Dichroism (XMCD/XMLD) and PhotoEmission Electron Microscopy (PEEM). Over the last year, our research community has used the polarised x-ray probes to explore a wide range of advanced materials to discover fascinating new phenomena. In this contribution, we present research demonstrating how REXS on I16 and I10 can reveal the long-range ordering of vortex domains in ferroelectric materials. On I21, RIXS has been used to discover electronic ordering in the parent compound of a new class of superconducting materials, further establishing the similarity between Mott insulators, such as the cuprates, and nickelates. On I06, a Mott insulator was imaged through its metal-insulator transition using time-resolved PEEM demonstrating the possibility of electronically generating new, non-equilibrium metallic phases of matter following ultrafast optical pulse excitation.

Ferroelectric materials play a key role in low-power nanoscale electronic devices and are commonly used in everyday applications such as sensors and actuators. The unique properties of these materials arise from their non-centrosymmetric crystal structure which enables electric polarisation under stress and vice versa. In recent years, topological vortex structures have been discovered in ferroelectric $PbTiO_3/SrTiO_3$ superlattices using high-resolution Transmission Electron Microscopy (TEM) which revealed picoscale swirling atomic displacements in thinned samples. On I10, these $PbTiO_3/SrTiO_3$ superlattices were further investigated using polarised soft x-ray REXS to map the long-range distribution and structure of the vortices. The study explored the three-dimensional structure of the polar vortices by comparing circularly polarised light scattering with theoretical simulations developed at Diamond. The combined study allowed a detailed 3D picture of the chiral electric polarisation structures to be built for the first time. On I16, ferroelectric thin films of $PbTiO_3$, sandwiched between $SrRuO_3$, were investigated using the power of hard x-ray REXS. Reciprocal space maps recorded on I16 were combined with high-resolution TEM and theoretical calculations to demonstrate the presences of long-range vortex structures in the sandwiched $PbTiO_3$ thin films which were interpreted as arising from the electric analogue of the magnetic Dzyaloshinskii–Moriya interaction.

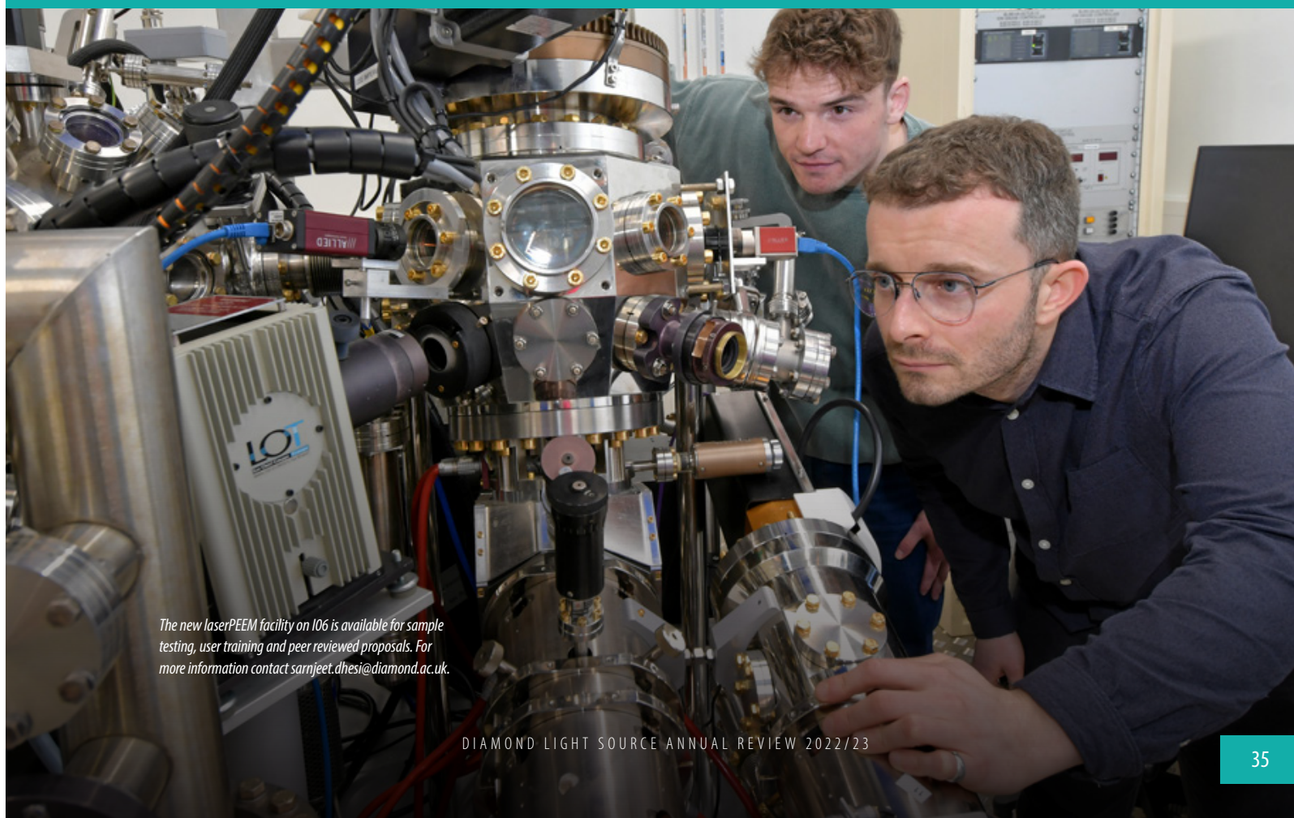
Mott insulators have very little energy difference between different phases so that magnetic fields, optical pulses and temperature can be used to drive such materials across electronic and magnetic phase boundaries. On I06, ultrafast optical pulses have been used to launch a metal-to-insulator transition in V_2O_3 with PEEM imaging showing that a new metastable, metallic phase retains nanoscale monoclinic distortions usually associated with the insulating phase. The aim in the future is to combine ultrafast optical pulses with electric fields to achieve low-power, reversible control of the metal-to-insulator transition. Doped Mott insulators have been studied extensively to understand the pairing mechanism in high-T_c superconducting materials such as the cuprates. Decades after the discovery of high-T_c superconductivity, the pairing mechanism and the influence of charge-density wave order remains ill-understood. On I21, the parent compound of infinite-layer nickelate superconductors has been investigated using RIXS. Interestingly, the charge-density waves competing or cooperating with superconductivity in the cuprates is found to exist in the layered nickelates too. The work indicates that, despite the differences between cuprates and nickelates, similar underlying electronic states and layered structures are important for superconductivity.

In the past year, the MMG has undergone an international review with several commendations regarding the capabilities of the team and the instruments. The review panel recommended that the MMG develop new sample environments, new sample management systems, wider data analysis workflows and that programmatic science themes across the beamlines should be explored and developed. In parallel, a range of recommendations were made to upgrade the beamlines as well as implementing new capabilities such as coherent diffraction imaging. In this sense, the MMG has been busy improving its suite of instruments. Beamline I21 now operates at a higher energy range (~ 3 keV) giving access to complex ferroelectric and magnetic ordering in *4d* transition metals. Recently, a new multilayer grating has been installed with a factor >30 increase in the detected photon flux during the first RIXS commissioning experiments performed at the Ru L_3 edge. Future plans involve a second multilayer coating to cover the S K-edge which will be pivotal to research into energy materials on I21. Beamline I06 has started user operations on the new laserPEEM facility with first experiments investigating local rotational orientations in 2D materials. The laserPEEM facility is equipped with a high intensity continuous wave laser allowing magnetic contrast imaging and also hosts surface preparation and characterisation facilities including Scanning Tunnelling Microscopy, Low Energy-Electron Microscopy (LEEM) and spatially resolved Low-Energy Electron Diffraction on the sub-micron scale. In the past year, the laserPEEM has been used to train the user community in the principles of PEEM and LEEM via one day workshops comprising lectures and hands-on practical sessions. On I16 a new low-vibration cryostat is being commissioned for REXS studies of micron sized samples and on I10 an in-plane, rotatable magnetic field for the RASOR diffractometer is being developed. The MMG has also established a Materials Characterisation Laboratory (MCL) to screen and align samples ahead of beamtime. The laboratory hosts a SuperNova Diffractometer, VSM SQUID, Atomic Force Microscope, Magnetic Force Microscope and sputter deposition facilities. The new Diamond-II Coherent Soft X-Ray Imaging and Diffraction (CSXID) flagship beamline has been the focus of considerable activity over the last year with the user working group reviewing the technical design report and plans to clear the area around straight I17 being progressed. In the coming year, the MMG will focus on harmonising the user experience across the beamlines and explore new strategies for sample management and metadata structures that further enable automated analysis.

The Materials Characterisation Laboratory (MCL) hosts facilities for x-ray diffraction, imaging and magnetometry. For more information contact sahil.tippireddy@diamond.ac.uk.

"The Magnetic Materials Group enables competitive, world-leading research in the UK."

(Findings of the International Review of the Magnetic Materials Group)



The new laserPEEM facility on I06 is available for sample testing, user training and peer reviewed proposals. For more information contact sarnjeet.dhesi@diamond.ac.uk.

Using light pulses to control electronic properties in vanadium oxides

Quantum Materials – Hard Condensed Matter – Electronic Properties – Physics – Materials Science – Imaging

Vanadium(III) oxide (V_2O_3) is an interesting material that has been extensively studied due to its unique properties. At low temperatures, V_2O_3 is a Mott insulator, which is an insulator that is driven by strong electronic correlations.

However, at approximately 170 K, V_2O_3 undergoes a transition from an insulator to a metal, both structurally and electronically. This transition is fascinating as it raises questions about the fundamental mechanisms behind the change. Is it purely electronic, or does a change in symmetry drive it? Answering these questions is crucial to understand the properties of V_2O_3 fully.

One of the exciting things about the insulator to metal transition is that it can also be induced by applying an electric field, which is known as resistive switching, or by exciting the material with ultrashort light pulses. This opens up the possibility of manipulating the conductivity of V_2O_3 on demand, which is essential for developing non-linear devices and neuromorphic materials. However, the transition speed is limited by the fact that, in quasi-equilibrium conditions, the electronic transition is always accompanied by a slower lattice change.

Recent research has proposed that ultrafast light excitation can drive a non-thermal transition that decouples the lattice change from the electronic one. This would allow researchers to control the phase transition on extremely fast timescales, enabling the material to transform from an insulator to a metal at frequencies as high as a few THz. This possibility offers tremendous potential for developing ultrafast Mottronics capable of operating at extremely high frequencies.

To study this phenomenon, an international team of researchers first demonstrated that the low-temperature insulating state of V_2O_3 is strongly inhomogenous. In contrast, the metalic phase is homogeneous at the nanoscale due to the lack of monoclinic distortion in the a-b plane. The team then took snapshots of the inhomogeneous nanotexture during the photo-induced insulator-to-metal phase transition using time-resolved X-ray Photoemission Electron Microscopy (PEEM) measurements at the I06 beamline.

The experiment demonstrated that the excitation with ultrashort near-infrared light pulses triggers the formation of a non-thermal state characterised by the electronic properties of the metallic phase, but retaining the same monoclinic distortion and nanotexture of the insulating state.

Their results unveil a profound and general link between the real-space topology, the transition dynamics and the emergence of non-thermal electronic states in quantum materials. Furthermore, the team's findings suggest possible routes to control metastable metallicity via the topology of the nanotexture.

By combining real-space morphology control via interface engineering, electric fields, or pressure with novel excitation schemes to coherently manipulate insulator-to-metal phase transitions, researchers hope to achieve full and reversible control of the electronic properties of correlated oxides.

In addition, the existence of light-induced non-thermal states in quantum materials is of great interest for resistive switching and neuromorphic computing applications. The ultimate goal is achieving all-electronic switching for ultrafast Mottronics capable of operating at frequencies as high as a few THz.

Related publication:
Ronchi, A. I Nanoscale self-organization and metastable non-thermal metallicity in Mott insulators. *Nature Communications* **13**, 3730 (2022). DOI: 10.1038/s41467-022-31298-0

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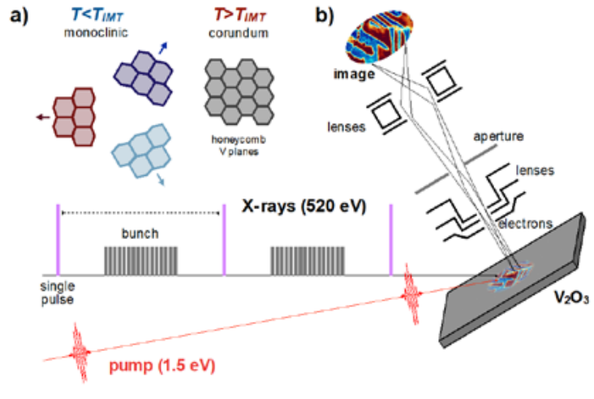

*a) **Monoclinic distortion**. Cartoon of the distortion of the hexagonal lattice along one of the three symmetry axes. Above and below the insulator to metal transition temperature, T_{IMT}*

*b) **Experimental setup**. X-rays bunches with tuneable energy resonant with the vanadium $L_{2,3}$ edge impinge on the sample. The electrons emitted from the sample are collected and imaged through electron lenses. In the time-resolved configuration, the signal originated by isolated X-rays pulses with linear horizontal (LH) polarization is collected by suitable synchronized gating of the detection apparatus. The pump infrared laser is synchronized to the synchrotron pulses.*

X-ray resonant scattering reveals chiral structures of polar vortices in ferroelectric superlattices

Quantum Materials – Hard Condensed Matter – Electronic Properties – Physics – Materials Science – Multiferroics

Recent research discovered polar vortex domains consisting of polar dipole vectors in ferroelectric superlattices. Because of their high potential as next-generation memory devices or functional devices, it is important to understand in detail the structure of polar vortices because it affects their properties and how they can be utilised in devices. Resonant Elastic X-ray Scattering (REXS) offers a non-destructive method to understand the complete 3D structure of the vortex.

An international team of researchers used the soft X-ray scattering setup at Diamond's I10 REXS experiment to determine the distribution of the three-dimensional polar vectors formed in a $PbTiO_3/SrTiO_3$ superlattice. This setup allows the X-ray energy to be resonantly tuned to the L absorption edge of titanium, as well as providing polarisation control of the X-rays. The handedness of the circular polarisation of incident X-rays must be freely changed to measure X-ray Circular Dichroism (XCD), which is the difference between X-ray scattering intensities for left- and right-handed polarisation and allows X-ray resonant scattering to distinguish chiral structures.

The research team observed that the sign of XCD varies depending on the incident angle of the X-rays. A new resonant scattering theory for quantitative analysis of the results was also required. Using a newly developed quantitative calculation, they revealed that this result meant a three-dimensional vortex array structure, not a simple one-dimensional helix.

Their research results are expected to play a significant role in the understanding and application of ferroelectric chiral domain structures. This method is non-destructive and uses resonant scattering, which enables real-time observation of subtle changes due to external stimuli such as electric fields or high-power laser pumping at ultrafast time scales.

This research on the polar structure of ferroelectrics corresponds to the counterpart of X-ray resonant magnetic scattering for the magnetic structure, which has already been extensively studied. These similarities naturally trigger X-ray resonant scattering studies on multiferroics that exhibit both properties at the same time. There is still no way to simultaneously measure two properties of the same atom or system, and X-ray resonant scattering is likely to be the answer.

Related publication:
Kim, KT. *et al.* Chiral structures of electric polarization vectors quantified by X-ray resonant scattering. *Nature Communications* **13**, 1769 (2022). DOI: 10.1038/s41467-022-29359-5

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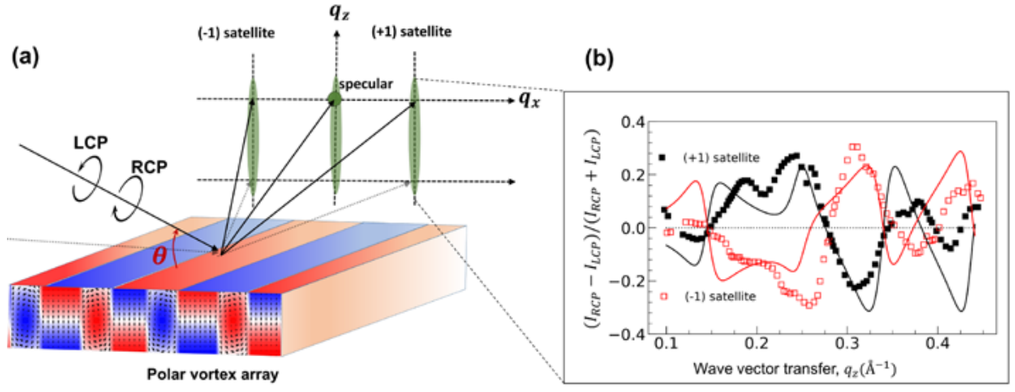

(a) Schematics of soft x-ray resonant scattering from polar vortex array. X-ray circular dichroism (XCD) intensities were measured along the qz direction at satellite peaks diffracted from the vortex array period. (b) XCD asymmetry ratios as a function of q_z proportional to the incident angle. Black and red symbols (lines) represent the measurements (calculations) at the +1 and -1 satellite peaks, respectively.

Ferroelectric topological structures

Quantum Materials – Hard Condensed Matter – Electronic Properties – Physics – Materials Science – Multiferroics

Although ferromagnetism and ferroelectricity are similar effects, they were thought to be fundamentally different. However, theoreticians suggested that we might see that the two phenomena are surprisingly similar if we could get a close enough look. In ferromagnetism, the Dzyaloshinskii–Moriya interaction (DMi) gives rise to effects such as skyrmions, which are potentially very useful for next-generation electronic devices (spintronics). If an analogous mechanism were present in ferroelectric materials, it would offer intriguing possibilities for future applications.

Ferromagnets and ferroelectrics are both ferroic systems characterised by an ordering parameter (magnetisation and polarisation, respectively), aggregating at macroscopic levels into domains that can be switched by applying an external respective field. Under particular conditions, the ferromagnetic order parameter can assemble into complex topological patterns that are currently the subject of intense research as they hold substantial promise for improved devices.

According to textbooks, such complex structures should never occur in ferroelectrics. Nevertheless, the dielectric polarisation in a carefully grown single crystal ultrathin film heterostructure consisting of a ferroelectric layer of lead titanate ($PbTiO_3$) sandwiched between strontium ruthenate ($SrRuO_3$) layers assembles into a two-dimensional array of electric dipole vortices.

A team of researchers from the University of Warwick used cutting-edge imaging techniques to carefully analyse this pattern. They determined that the polarisation of the $PbTiO_3$ layer is also ordered along the third dimension, yielding a corkscrew-like pattern that is either helical or cycloidal and modulated in two orthogonal directions. Such a spacially ordered polarisation has not previously been observed in ferroelectric materials. The data support a model that proposes these complex polarisation patterns are driven by a mechanism similar to the DMi in magnetic materials.

To fully understand the coupling mechanisms, the team needed to study both the intra- and inter-layer structures across the atomic and meso (tens of nanometer) length scales. To ensure generality in the results, non-destructive and spatially averaged data such as that gleaned from X-ray diffraction was required.

They used Diamond's I16 beamline to perform high-resolution X-ray diffraction. The high resolution and high flux coupled with area detectors enabled them to study periodicities in reciprocal space with necessarily high resolution.

The X-ray data showed periodicities in two orthogonal directions that are aligned with the orthorhombic crystal symmetries. The X-ray data are in complete agreement with the more myopic cross-sectional Transmission Electron Microscopy (TEM) and show that the observed topology indeed extends throughout the crystal.

In the studied sample, the topologies arise from small tilts of the atomic positions within the crystal, which induce both a small electrical polarisation and strain. The observed ferroic topologies arise as the system attempts to minimise the internal energy. The observation of an electrical equivalent of the DMi shows that complex topologies of the polarisation are now possible. By changing the material properties, this interaction strength can be tweaked, driving new, ever more complex structures that can be stabilised. The plethora of technologies based on ferromagnetic spin textures shows what may be possible in these electrical equivalents.

Related publication:
Rusu, D. *et al.* Ferroelectric incommensurate spin crystals. *Nature* **602**, 240–244 (2022). DOI 10.1038/s41586-021-04260-1

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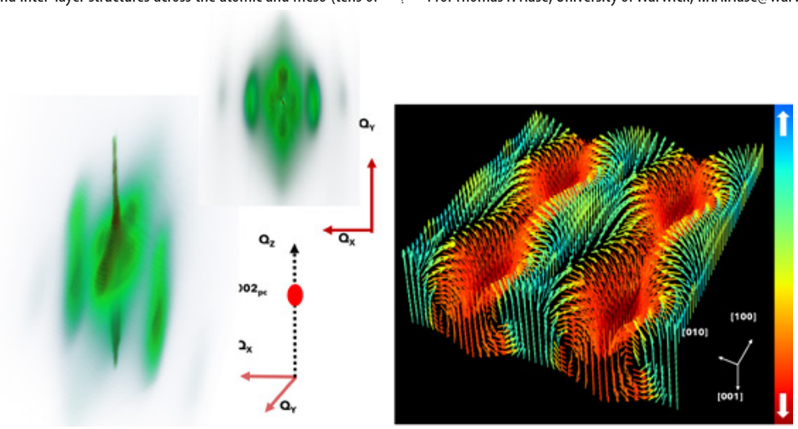

Reciprocal Space Volume reconstructed from a series of 2D images taken during a rocking curve of the (002) substrate Bragg peak in $DyScO_3$ (left). Projection onto (Qx – Qy) wave vectors, giving a Reciprocal Space Map (RSM) around the same reflection (middle-top). The derived model of the Dielectric polarization pattern in the lead titanate (PTO) showing polarisation curling assembled in periodic vortices and a second modulation consisted of a cycloidal twist of electric dipoles along the axis of the polar vortice s (right). Red colour indicates a downward polarization direction, blue -upwards, and yellow -horizontal.

Charge-ordered phase in layered nickelates

Quantum Materials – Hard Condensed Matter – Electronic Properties – Physics – Materials Science – Superconductors

Unconventional high-temperature superconductors are at the core of quantum materials and advanced technological applications. Scientists have been trying to synthesise new superconductors with ever-higher superconducting critical temperatures and to unlock the mechanism underlying the zero-resistivity behaviour. Recently a new family of superconductors with layered nickelate-oxide structure was successfully obtained. The latest resonant X-ray spectroscopic studies reveal them to be very close cousins to copper-oxide superconductors (cuprates) and highlight the importance of the strong electronic Coulomb interaction, the quasi-two-dimensional electronic structure of the MO_2 (M=Cu, Ni) building block, and the strong antiferromagnetic magnetic excitations.

All these suggest we may have found the right recipe for producing a high-temperature superconductor. However, significant challenges are to be urgently solved. For instance, so far, superconductivity only exists in thin films of layered nickelates, and less than a dozen research groups could successfully synthesise them, prompting a rather unstable state of the material. Moreover, scientists are still striving to understand why their superconducting critical temperature is about an order of magnitude lower than that of cuprates ($T_c \sim 130\ K$).

In cuprates, doped electron or hole charges, besides forming Cooper pairs in the superconducting state, may segregate into regions with periodically variable density, *i.e.* forming charge-ordered phase. So, naturally, we may wonder whether such charge-ordered phases exist in their close cousin, layered nickelates.

An international team of researchers addressed this question using Diamond's I21-RIXS beamline. Resonant Inelastic X-ray Scattering (RIXS) is one of the few techniques capable of probing the charge-ordered state. In particular, owing to the sub-100 nm probing depth, RIXS is particularly suited for the nm-thick nickelate films. Moreover, as I21 is equipped with high energy resolution and high photon flux, it is an ideal facility for this type of study.

The research team found the charge-ordered states exist in the parent layered nickelate $NdNiO_2$. They show strong resonances for both the Ni 3*d* and Nd 5*d* states, illustrating a coupled electronic structure between the two. The charge-ordered state also shows a clear temperature dependence, a hallmark of an ordered state with an electronic origin. The continuous tunability of the RIXS spectrometer was then used to show that the charge-ordered state has a non-negligible *L*-dependence, hinting that it is a three-dimensional object.

Despite the differences to cuprates, the existence of the charge-ordered state demonstrates that the layered nickelates are remarkably similar to cuprate superconductors. It is clear that the combination of the MO_2 (M=Cu, Ni) building block, an active dx^2-y^2 orbital near Fermi level, and spin S=1/2 antiferromagnetic (AFM) correlation, are crucial for achieving unconventional superconductivity. Understanding the common collective excitations of the AFM correlation and the charge-order may provide essential knowledge to eventually unfold the superconducting pairing mechanism.

Many materials in condensed matter physics exhibit remarkable properties such as zero-resistivity, colossal magneto-resistance and the magneto-optical Kerr effect. These novel properties lie in the competition of electronic and magnetic interactions under the angstrom and nanometre scale. A spectroscopy technique such as RIXS is imperative to understand and eventually make newer materials with richer functionality, due to its remarkable sensitivity.

Related publication:
Tam, CC. *et al.* Charge density waves in infinite-layer NdNiO2 nickelates. *Nature Materials* **21**, 1116–1120 (2022). DOI: 10.1038/s41563-022-01330-1

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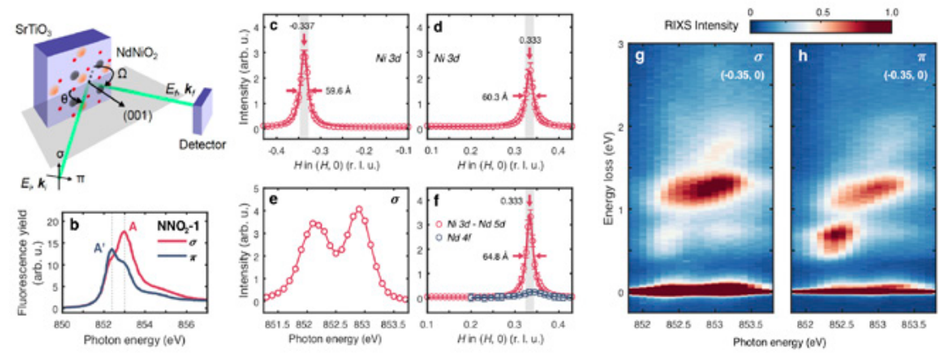


a, The experimental geometry of XAS and RIXS experiment. b, Ni L_3 XAS of the parent $NdNiO_2$ film. c,d, Integrated quasi-elastic peak intensity as a function of momentum transfer along the (±H, 0) direction by excitation at the Ni 3d resonance (A peak in b). e, The resonant energy profile of the charge-order at (+0.333, 0). f, Integrated quasi-elastic peaks as a function of momentum transfer along the (H, 0) direction by probing the Ni 3d–Nd 5d hybridized state (A' peak in b) and Nd 4f states at the Nd M_5 edge. g,h, RIXS maps on layered nickelate at 20 K at H = −0.35 r.l.u. with σ (g) or π incident X-ray polarisation (h).

Imaging and Microscopy Group

Paul Quinn, Science Group Leader

The Imaging and Microscopy Group brings together eight experimental facilities (I08, I08-1, DIAD, I12, I13-1, I13-2, I14 and ePSIC) which use electrons and X-rays to image samples under different experimental conditions across a diverse range of length scales and time scales.

The DIAD beamline for Dual Imaging and Diffraction offers two X-ray techniques, full-field radiography/tomography and micro-diffraction, used on the same sample quasi-synchronously. This setup enables in-situ characterisation of the 3D microstructure of the material at the same time as its crystallographic phase and/or strain state. X-rays from a 10-pole wiggler are split into two independent beams and then combined at the sample position. Imaging beam can be operated in either pink or monochromatic mode; diffraction is conducted with monochromatic mode. Both beam energies can be chosen independently of each other in an energy range of 8-38 keV. DIAD expects the delivery of a dedicated mechanical test rig with integrated tomography capabilities which will be an integral part of the end station and enable a variety of scientific experiments in engineering, materials science, biomaterials and hard tissues, geology and mineralogy. DIAD is part-funded by the University of Birmingham.

The Scanning X-ray Microscopy (SXM) beamline (I08) is for morphological, elemental and chemical speciation on a broad range of organic-inorganic interactions in a 250 - 4400 eV photon energy range, and sample investigations under ambient or cryogenic conditions. I08 has a range of applications including biological and biomedical sciences, earth and environmental science, geochemistry, and materials science. I08 improved and partially automated and simplified User operation. The new soft X-ray spectro- and tomo-ptychography branchline will provide a step change in imaging and spectro-microscopic performance for soft X-ray imaging at Diamond. The branchline is growing in capabilities and has been enthusiastically received by initial users (I08-1 took first users in Oct 2020) with clear benefits for experiments for many experiments transitioning from I08 to I08-1. Key developments for I08-1, such as providing access to the carbon edge for ptychographic studies are well under way and developments to deliver tomographic and cryogenic capabilities are also planned for the next year. Applications for user experiments are now accepted through our standard call.

The Joint Engineering, Environmental and Processing (JEEP) beamline (I12) uses a 4.2 T superconducting wiggler to provide polychromatic and monochromatic X-rays in the energy range 53 to 150 keV. These high photon energies provide good penetration through large or dense samples. The beamline offers beam sizes ranging from 50 x 50 μm^2 for diffraction, up to 90 x 25 mm^2 for imaging and tomography. Static objects larger than the available beam can be tomographically imaged using special scanning protocols. The beam characteristics enable the study of macroscale samples that are representative of bulk materials and processes. Another feature of I12 is the ability to use complex, enclosed sample environments without unacceptable attenuation of the beam. X-ray techniques available are radiography, tomography, energy-dispersive diffraction, monochromatic 2D diffraction and scattering. Radiography and tomography are performed predominantly with monochromatic X-rays. Polychromatic beam is reserved for energy-dispersive diffraction or non-routine special requests. I12 has a diverse user community (materials science & engineering; chemical processing; biomedical engineering; geoscience; environmental science; physics; palaeontology) who make full use of the beamline's capabilities. The beamline's two flexible experimental hutches allow users to bring their own rigs and sample chambers. I12 continues to support a wide range of *in situ*, time resolved experiments, notably in additive manufacturing, materials property testing and chemical processing. It is common for users to combine imaging and diffraction in the same experiment. Almost all tomography scanning is done with constant speed stage rotation, to reduce scan times.

The I13 Imaging and Coherence beamline is for multi-scale imaging in the energy range of 6 -30 keV. The achievable resolution ranges from several microns to some tens of nanometers with two branchlines operating independently for this purpose. The Diamond Manchester Imaging branchline performs mainly in-line phase contrast tomography with a strong emphasis on dedicated sample environments. A full-field microscope using Zernike phase contrast imaging over a field of view of 50-100 µm and a resolution of 50 - 100 nm is in operation, with a growing user community, allowing us to identify nano-sized structures under dynamic conditions. A broad set of user groups are now exploiting the robot arm facility for static samples and high-throughput studies. The highest spatial resolution, of 30 nm, is achieved on the coherence branch with ptychographic imaging. Ptycho-tomography scans can be performed on a time scales of hours and this is enabling new spectroscopic and dynamic experiments. Bragg-CDI and new developments in Bragg Ptychography provide complementary experimental capabilities for studying nano-crystalline structures .

I14, the Hard X-ray Nanoprobe beamline, offers a beam of 50 nm for high resolution imaging. I14 has expanded the core 2D techniques of X-ray fluorescence, diffraction, X-ray Absorption Near Edge Structure (XANES), differential phase contrast and ptychography for mapping inhomogeneities in a wide range of samples to include tomography for volume imaging of elemental, structural and chemical states. A wider range of complex in-situ experiments in a range of conditions such as electrochemistry, liquid and gas environments have been enabled by dedicated effort to expand the sample cells available and optimise the measurement conditions. .

The electron Physical Science Imaging Centre (ePSIC) at Diamond consists of two transmission electron microscopes, a JEOL ARM 200 and a JEOL GRAND ARM 300, which were brought to Diamond through a collaboration with Johnson Matthey and the University of Oxford respectively. The ARM 200 is a state-of-the-art probe-corrected analytical microscope capable of atomic resolution electron energy loss and X-ray spectroscopy. The ARM 300 is a dedicated imaging instrument aligned across a wide range of accelerating voltages (30 - 300 keV) and is equipped with an Oxford Instruments X-Max 100 EDX detector. It is both probe- and imaging-corrected and has numerous detectors, including a small pixel array (512 x 512) fast direct electron detector for low voltage work and a newly installed large pixel array (4K x 4K) fast direct electron detector for high voltage imaging. These combined capabilities make this a unique resource for electron microscopy within the UK. With *in situ* sample holders, users at ePSIC can perform variable temperature measurements from 100 to 1600 K, apply electrical bias to samples during imaging and transfer samples anaerobically into the microscope. For TEM sample preparation ePSIC runs a JEOL 4700F focused ion beam microscope with in-situ lift out and anaerobic transfer capability. The state-of-the-art instrumentation available at ePSIC attracts both established electron microscopists looking to develop new techniques, and scientists with limited previous electron microscopy experience interested in the atomic structure of their samples. ePSIC is moving to a 3-month peer review cycle to be able to respond more quickly to science needs and to also provide a mechanism to improve correlative experiments, enabled by the unique co-location of ePSIC at Diamond.

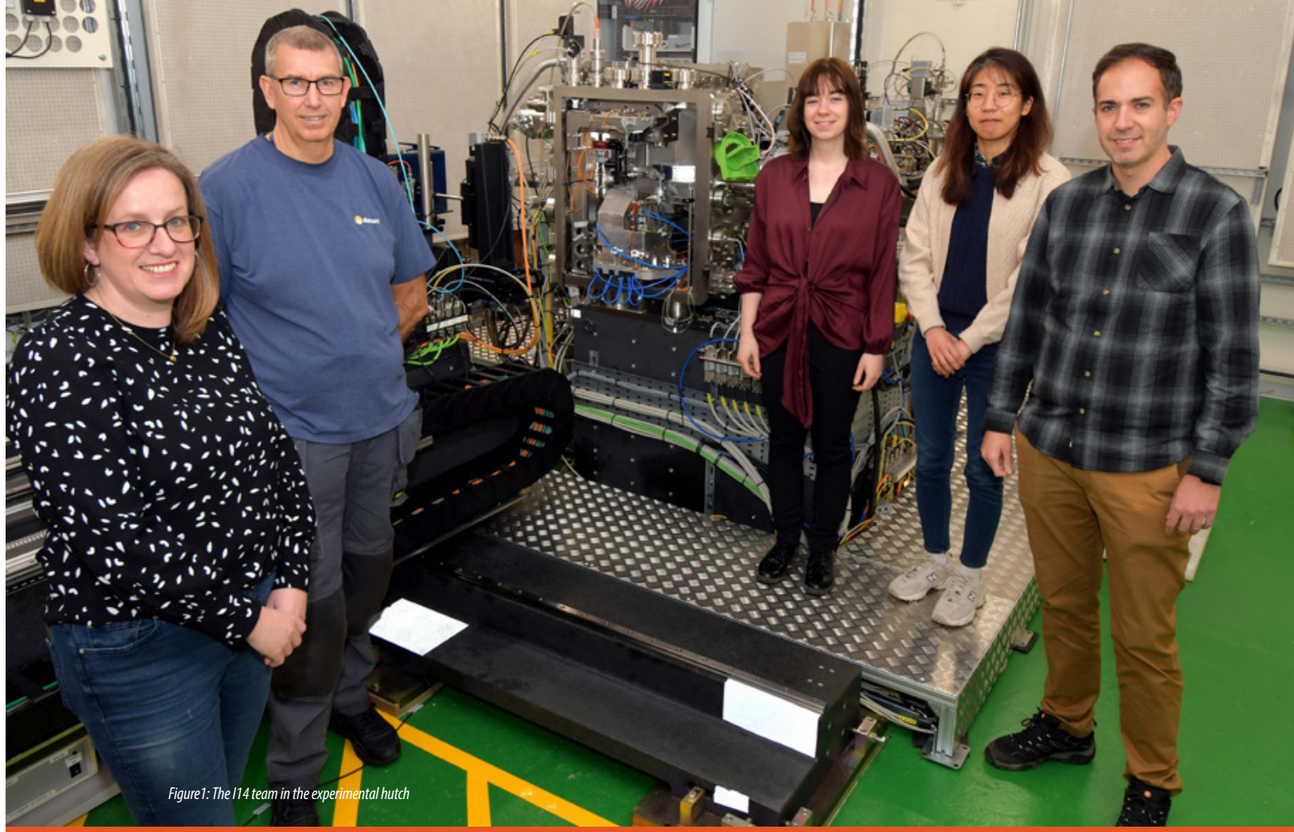
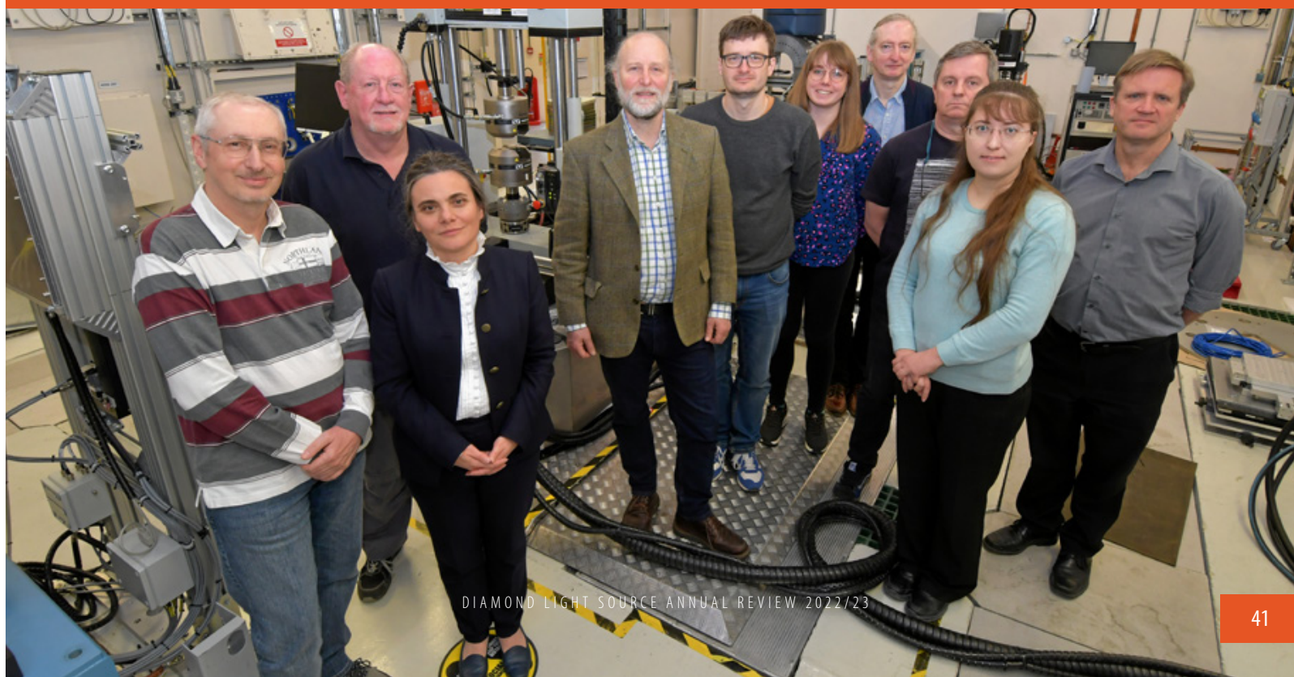


Figure 1: The I14 team in the experimental hutch

"ePSIC is moving to a 3-month peer review cycle to be able to respond more quickly to science needs and to also provide a mechanism to improve correlative experiments, enabled by the unique co-location of ePSIC at Diamond"

Figure 2: I12 team and support staff on the beamline (experimental hutch 1 and 2).



Searching for life and for our origins are two sides of the same coin

Earth Sciences & Environment – Geology – Geochemistry – Planetary Geology

In 1996, researchers at NASA Johnson Space Center released a paper in Science entitled "Possible relic biogenic activity in Mars meteorite ALH84001". This caused intense scientific examination of this meteorite, ultimately leading to the formation of the field of Astrobiology and renewed interest in sending missions to Mars. After several years, the scientific consensus was that there was no evidence of Martian life in the meteorite, but the question of organic material remained ambiguous. So the question remained for many years: if ALH84001 does not contain signs of martian life, what is the nature of organic material in this meteorite, and what is its origin?

An international team of researchers undertook a series of investigations to spatially resolve the presence, nature and possible synthesis mechanisms for the organic material in ALH84001. They used a Focused Ion Beam (FIB) instrument to cut sections from the matrix and carbonate rosettes within the meteorite and performed Transmission Electron Microscopy (TEM), Scanning Transmission X-ray Microscopy (STXM) and D/H measurements on those sections. That enabled them to find the organic carbon and study its provenance and relationship to minerals in the meteorite.

At Diamond's I08 beamline, they carried out high spatial resolution STXM analysis across the C and N edges to look at the presence and bonding environment of the carbon present. The results revealed a high proportion of aromatic and oxygen functionality in the organic material. Finally, after completing these analyses, they measured the hydrogen isotopic signal of the analysed organic carbon to ensure its martian origin.

From these analyses, the team were able to show that the organic carbon was synthesised *in situ* on Mars in two processes: serpentinisation and carbonation. These two organic synthesis mechanisms had not been seen on Mars before and are indicative of water-rock reactions 3.6 billion years ago. Water-rock interactions are relevant to planetary habitability, influencing mineralogical diversity and the production of organic molecules.

This discovery has enabled them to set a non-life background for the detection of a non-terrain-based life form but also points to fundamental reactions between igneous rocks and brines that lead to the synthesis of abiotic/prebiotic organic material that produced life on early Earth. While the record of these interactions on early Earth has been destroyed by plate tectonics and overprinted by life on Earth, that record appears intact on Mars.

Related publication:
Steele, A. *et al.* Organic synthesis associated with serpentinization and carbonation on early Mars. *Science* **375**, 172-177 (2022). DOI: 10.1126/science.abg7905

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Meteorite fragment ALH84001 found in Antarctica in 1984, Wikimedia commons.

Paving the way for stronger alloys

Materials Engineering & Processes – Materials Science – Engineering & Technology - Metallurgy

Understanding the formation of microstructures is critical to a wide range of solidification procedures, including casting and welding. Research conducted by scientists at the University of Birmingham has expanded our understanding of alloys by exploring how microscopic crystals evolve in molten metals as they cool. Their breakthrough research has the potential to enhance the tensile strength of alloys utilised in aerospace and automobile applications.

A microscopic examination will show faceted dendritic intermetallic crystals in all sorts of metal alloys, including nickel-based superalloys, steels and aluminium alloys. Although these crystals significantly affect the properties of alloys, there is a scarcity of research dedicated to studying their formation mechanisms.

The classic view of dendrite formation is that as a spherical solid nucleus in undercooled liquid melt grows, the solid-liquid interphase becomes unstable. Perturbations form consequently, leading to the formation of dendrites. The research team was interested in whether this mechanism applies to the formation of faceted intermetallic dendrites.

They built a furnace to melt an aluminium-copper (Al-Cu) alloy and control the cooling, and coupled it with high-speed tomography at Diamond's I12 beamline. This setup allowed them to capture 'snapshots' in seconds, resolving the topological evolution of the crystals as they formed at high temperatures.

The intermetallic Al_2Cu in the Al-Cu alloy forms in many shapes, from simple rods to complicated faceted dendrites. The study shows that, in a cooling aluminium-copper alloy, the solidification processes with the formation of faceted dendrites. These dendrites form by a layer-by-layer stacking of small, micrometre-sized basic units. These units start L-shaped and stack on top of each other like building blocks. However, they change shape as they cool, first transforming into a U shape and finally a hollowed-out cube, while some stack together to form beautiful dendrites.

These findings provide new insights into what happens at a micro level as an alloy cools, and show the shape of the basic building blocks of crystals in molten alloys. The results directly contrast with the classical view of dendrite formation in cooling alloys, opening the door to developing new approaches to predict and control intermetallic crystal formation. As crystal shape determines the strength of the final alloy, if we can make alloys with finer crystals, we can create stronger alloys. Dendrites also form in processes other than the solidification of metals. It's possible that this mechanism may also explain the formation of dendrites in cooling magmas and cycling batteries.

Related publication:
Song, Z. *et al.* Revealing growth mechanisms of faceted Al2Cu intermetallic compounds via high-speed Synchrotron X-ray tomography. *Acta Materialia* **231**, (2022). DOI: 10.1016/j.actamat.2022.117903

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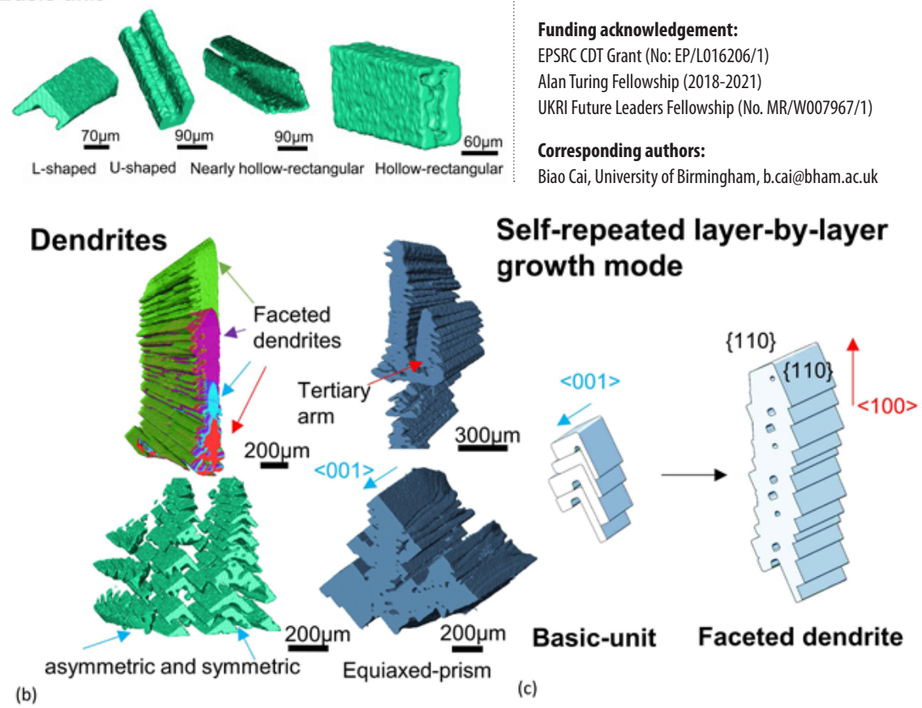

(a) the basic units of Al_2Cu intermetallic compound, (b) dendrites of Al_2Cu, and (c) the layer-by-layer growth mechanism for Al_2Cu dendrites.

Detailed measurement of spinal disc mechanics provides insight into common back pain

Health & Wellbeing – Life Sciences & Biotech

The leading cause of years lived with disability is from people suffering with lower back pain. It severely affects the quality of life of all global populations. The spine (intervertebral) discs are the soft tissue in the spinal column. When injured or degenerated, they are responsible for the majority of these lower back pain cases. Currently, only symptomatic treatment or spinal surgery is available; therefore, understanding the mechanical pathogenesis of degeneration is crucial to developing non-invasive therapies.

Tissues are developed, maintained, and repaired through the actions of cells using signals from their micromechanical environment, but the ultimate function of large orthopaedic structures such as the spine is mechanical – providing both strength and flexibility to the core of our bodies. This system is elegant but also prone to degradation and injury. The development of better medical approaches to spine problems will continue to be elusive without a proper understanding of tissue mechanics at the level of the cell.

Despite a wealth of clinical evidence, we do not yet know whether there is a regional distinction in collagen fibre architecture and whether deformation under mechanical load is region-specific. Therefore, researchers used synchrotron Computed Tomography (sCT) on Diamond's I13-2 beamline to investigate collagen fibre bundles in 3D throughout an intact native rat lumbar IVD under increasing compressive load.

Spinal discs are large structures and must be kept intact to preserve mechanical characteristics. Still, the outer disc material is an intricate collagen fibre composite requiring high-resolution (micron scale) visualisation. Furthermore, unlike the adjacent vertebrae, spinal discs are unmineralised (with no bone tissue), so standard X-ray methods are unsuited. Add to this the need for compression loading to simulate spine function, and a combination of I13-2 beamline capabilities becomes essential: soft tissue imaging (phase contrast), applying load whilst imaging (*in situ* tomography), and imaging volumes large enough for animal spine models (millimetres to centimetres).

By directly examining the functional response of intact spinal discs, in high enough detail to observe the critical collagen fibres, this study contributed to the understanding of disc degradation and failure. Portions of the disc that fail most frequently (posterior-lateral) have an inherently different fibre architecture from regions less prone to failure (anterior) and have a lower capacity to respond to compressive loading. The level of detail in these observations, with tens of thousands of individual fibres visualised and measured within each region, is essential to a full understanding of disc mechanics.

The structure and function patterns revealed in this study suggest a compromise between tissue flexibility and strength characteristics that is important to acknowledge when preventive and treatment therapies for disc pathologies are developed. They also provide a roadmap for attempts to restore spinal tissues to a more functional state. Being able to perform detailed characterisation of numerous individual collagen fibres from *in situ* imaging of intact tissues and structures will enhance our understanding of tissue biomechanics. This technique will allow us to discover the structures that cause some tissue regions to be more susceptible to degeneration. The study outlines a powerful methodology to evaluate the performance and guide the development of new treatments as they emerge.

Related publication:
Disney, CM. *et al*. Regional variations in discrete collagen fibre mechanics within intact intervertebral disc resolved using synchrotron computed tomography and digital volume correlation. *Acta Biomaterialia* **138**, 361-374 (2022). DOI: 10.1016/j.actbio.2021.10.012

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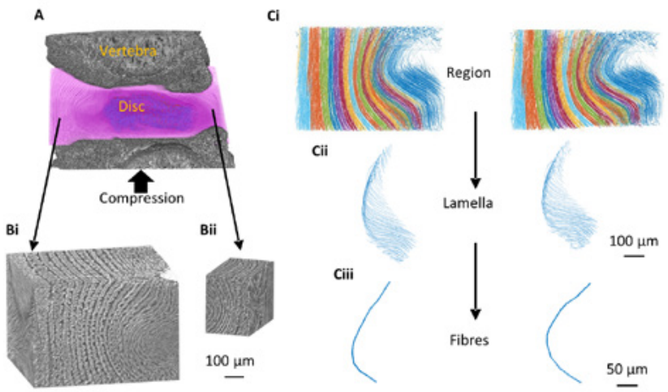

A: Three-dimensional imaging of a whole rat spine segment imaged at I13 where soft tissue microstructure could be resolved. Spine segments were compressed during measurements to record structural changes during mechanical function. B: Regional comparisons can be made within the same sample to determine structural-mechanical aspects that could lead to degeneration. C: Tissue biomechanics such as deformation measured as fibre strain and morphological changes was evaluated at three structural levels of region (i), lamella (ii) and individual fibre (iii). (Left column before the sample was compressed, right column after the sample was compressed).

High throughput ptychographic tomography at the I13 Coherence Branch

Technique Development – Materials Science

X-ray Ptychography is an imaging technique that scans a sample through a coherent X-ray beam to collect diffraction information from overlapping regions before reconstructing them into a high-resolution image. This technique produces quantitative phase images with the highest possible spatial resolutions, going well beyond the conventional limitations of the available X-ray optics, and has wide-reaching applications across the physical and life sciences. However, scanning techniques have inherent overheads, with the need to move sample stages and capture motor positions and detector frames. The data collection time for ptychographic tomography data was typically 24 hours, with only a fraction of that time actually exposing the detector. The problem was that the data collection overheads, caused by the time required for motor settling and file writing, were limiting the throughput of the imaging method. As a result, Ptychography has remained significantly slower than direct methods such as Transmission X-ray Microscopy (TXM) and Micro Computed Tomography (micro-CT), which limits its application to the wider scientific community.

Diamond scientists have taken steps to reduce the bottlenecks on the I13-1 beamline, making it the fastest ptychography beamline in the world. Faster scans enable the study of more dynamic and *in-operando* processes, increase the throughput of experiments and offer users real-time feedback.

Starting with a hardware scanning protocol created at I24, they began investigating continuous scanning strategies. Once in a continuous scanning modality, the scanning speed relates directly to the data collection rate. The fastest detector available for these measurements could run at up to 9kHz, but the motion controller at the beamline was limited to 800Hz. They investigated novel up-triggering and file-writing strategies that would permit rapid data collection.

The experiments and developments were performed and applied to the I13-1 coherence branch. The coherence branch performs multiscale and multimodal ptychographic tomography. This work opens up the powerful imaging capabilities available at the branchline to a much wider scientific community.

The approach they developed and implemented allowed for a 20µm^3 volume of battery material to be scanned at the nanoscale in under three hours. The work is continuing and is allowing for samples previously too large (>100µm^3) to be scanned in hours and smaller samples to be scanned in minutes.

A greater throughput of nanoscale ptychographic tomography allows us to study larger volumes of materials. In addition, statistically relevant data will be recorded. For example, understanding the functionality of brain tissue, requires imaging at the length scale of the synapses (10s nm) and connecting these pathways along several hundreds of microns. Also, studying smaller samples *in operando* at these length scales is now possible. That is exciting in many applications, for example, tracking the structural evolution within individual primary particles to understand battery degradation as the active material undergoes charging cycles.

A major milestone has been achieved by significantly accelerating the recording speed for tomography with ultimate X-ray resolution. As a result, significant numbers of samples can be investigated in three dimensions with resolution on the nanometre length scale.

Related publication:
Batey, D. *et al*. High-speed X-ray ptychographic tomography. *Scientific Reports* **12**, 1-6 (2022). DOI: 10.1038/s41598-022-11292-8

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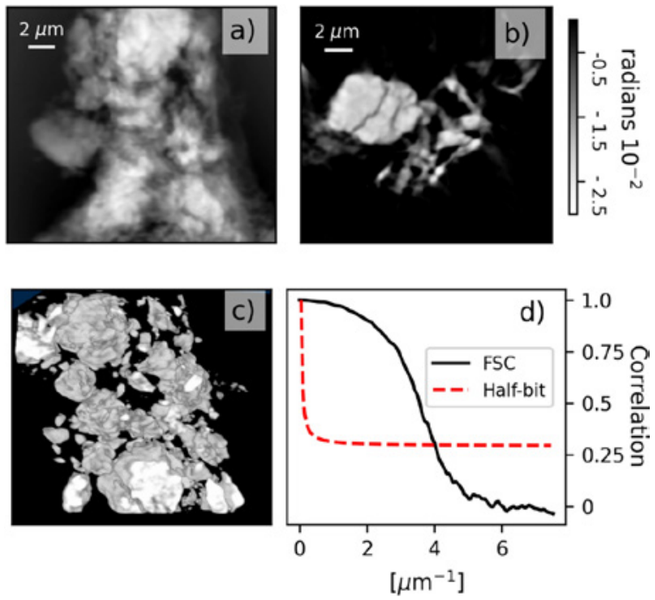

2 kHz ptychographic tomography scan reconstruction. (a) Reconstructed phase projection of NMC particle. (b) 2D slice of the 3D reconstructed volume. (c) 3D view of the reconstructed volume. (d) Fourier shell correlation for the 3D volume (resolution 250 nm).

Nanoscale impurities seed degradation in novel solar materials

Surfaces - Earth Sciences & Environment - Sustainable Energy Systems - Energy - Physics - Climate Change - Physical Chemistry - Energy Materials - Chemistry - Materials Science - Interfaces and Thin Films - Perovskites - Metallurgy

Perovskite materials offer a cheaper alternative to silicon for producing solar cells and also show great potential for other optoelectronic applications, including energy-efficient LEDs and X-ray detectors.

Metal halide salts - abundant and much cheaper to process than crystalline silicon - can be prepared in a liquid ink used to print a thin film of the material.

In the past decade, improvements in the design and fabrication of metal halide perovskite (MHP) based solar cells have seen their efficiencies rise to compete with incumbent technologies and have laid the pathway to commercialisation. However, MHP stability, and thus the longevity of these light-harvesting devices, remains deficient. A multidisciplinary team of researchers used Diamond's Hard X-ray Nanoprobe beamline (I14) and the electron Physical Science Imaging Centre (ePSIC) to gain new insight into the perovskite materials that hold so much potential in the field of optoelectronics.

Low-dose Scanning Electron Diffraction (SED) measurements performed at ePSIC allowed the team to map the crystallography of their MHP samples with 5 nm resolution at various stages of ageing under light exposure, without triggering additional electron beam-induced degradation. They also used complementary experiments at I14 to survey the various crystallographic structures present.

Their results showed that photochemical degradation of the MHP samples (manifesting as a change in crystal structure and eventual amorphisation) initially occurs in very localised sample regions. Crucially, these sample regions are crystalline grains or boundaries associated with unwanted material phases such as hexagonal polytypes and lead iodide - the same nanoscale structures that compromise light harvesting efficiency. The team concludes that degradation seeds at phase impurities due to their high density of defects, which act as both non-radiative recombination sites for charge carriers and fuel for fatal redox photochemistry. They uncovered one method of mitigating the formation of sinister hexagonal polytypes: controlled octahedral tilting of the perovskite lattice.

Their findings suggest that the localised presence of phase impurities are direct indicators of failure points in the absorber layer. The detection of such species through nanoscopic screening (e.g. high resolution electron microscopy) offers a means of predicting sites of instability during film optimisation and manufacturing for application in solar cells. There are several strategies for inducing beneficial octahedral tilt, including tuning the perovskite A-site cation or adding passivating organic molecules. New approaches should be developed to realise scalable, uniformly tilted and, thus, photo-stable MHP films on the manufacturing line.

This research could significantly accelerate the development of long-lasting, commercially available perovskite photovoltaics.

Related publication:

Macpherson, S. *et al*. Local nanoscale phase impurities are degradation sites in halide perovskites. *Nature* **607**, 294–300 (2022). DOI: 10.1038/s41586-022-04872-1

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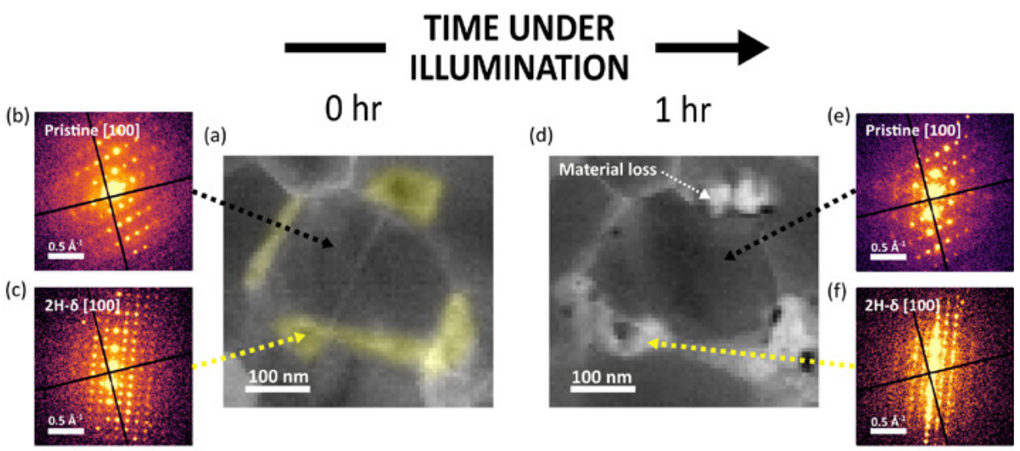

Scanning Electron Diffraction (SED) maps of nanoscale photodegradation on a metal halide perovskite thin film. *(a) Diffraction contrast image of fresh sample region before light exposure showing polycrystalline grain structure. Diffraction patterns identifying (b) a "pristine" perovskite grain and (c) hexagonal polytype perovskite phase impurities (highlighted yellow in (a)). (d) Diffraction contrast image of the same region after 1 hour of solar-equivalent light exposure. Light contrast regions are indicative of local material loss. (e, f) SED patterns from the same locations showing minimal evolution in the pristine grain, and loss of crystallinity in the polytype impurities.*

Nanoprobe and ePSIC analysis techniques for sample return missions

Earth Sciences & Environment – Geology – Geochemistry – Planetary Geology

Space weathering is a collective process that causes a gradual alteration in the composition, structure and optical properties of asteroids and other bodies that move through the solar system without a protective atmosphere.

The Hayabusa2 sample-return mission to near-Earth asteroid (162173) Ryugu provides the first opportunity for laboratory studies of space-weathering signatures on a C-type asteroid, the most abundant type of inner solar system body. C-type asteroids are composed of materials that have remained largely unchanged since the solar system formed.

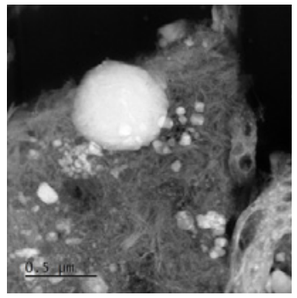

Figure 1: TEM image taken at E01 ePSIC showing Ryugu serpentine and Fe oxide minerals.

The Hayabusa2 mission was designed to bring back samples from two different types of site on Ryugu. One sample collection was made at the surface, and the other was in a small crater excavated by firing a copper projectile at the asteroid from the spacecraft. The aim of comparing excavated samples from the crater to surface samples was to identify the processes that control the spectral properties – how we classify asteroid types from Earth – and the surface mineralogy of asteroids.

The Hayabusa2 Earth Return Capsule landed in the Nullabor desert of Western Australia in 2020. Curated at the Japanese Space Agency Sagamihara Facility near Tokyo, samples were prepared for the international analytical team, including the University of Leicester, as a result of the preparatory work done at Diamond and ePSIC.

The research team performed scanning and high-resolution Transmission Electron Microscopy on the Ryugu grains at ePSIC. They also used Diamond's I14 beamline for nanoscale X-ray Absorption Spectroscopy and Fluorescence mapping on the same samples. Their results gave the most accurate determinations of the valency state of the iron in the outer rind of the Ryugu grains that had been exposed to space for millions of years.

From the textural and spectroscopy results at I14 and ePSIC, combined with work from Hayabusa2 mission colleagues in Japan and worldwide, it has now been established that the surface of Ryugu has undergone intense bombardment by micrometeorite grains, the solar wind and galactic cosmic rays. Together this is what we now recognise as 'space weathering'.

New sample return missions to asteroids, Phobos (Mars' largest moon and perhaps a captured asteroid), the Earth's moon and, most ambitiously of all, Mars, over the coming decade, are designed to provide new insights about the evolution of the Solar System that can't be gained from *in situ* analyses by landers and orbiters. However, the mass of returned samples is constrained, perhaps 40 drill tubes totalling 500 g from Mars. Synchrotron analyses will be essential to perform 3D spectroscopy on complex mineral assemblages at a microscopic scale for upcoming sample return missions.

In addition to wanting a better understanding of the processes that have formed and altered the surfaces of the Solar System's asteroids, including targets for new sample return missions such as Phobos, new concepts in space exploration of *in situ* resource utilisation - and even asteroid mining – require a better understanding of how remote spectra of asteroid surfaces relate to the composition of their interiors.

Related publication:

Noguchi, T. *et al.* A dehydrated space-weathered skin cloaking the hydrated interior of Ryugu. *Nature Astronomy* **7**, 170–181 (2023). DOI: 10.1038/s41550-022-01841-6

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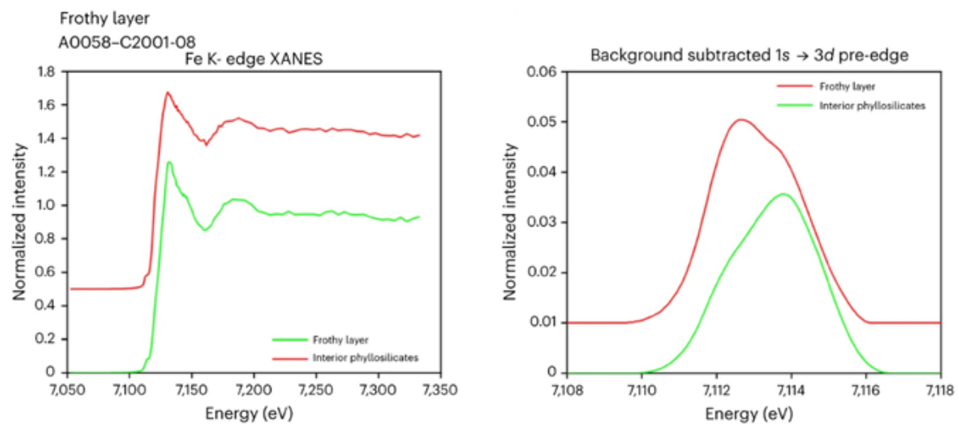

Figure 2: nano Fe XANES spectra from I14 indicating the Fe^{3+} to Fe^{2+} reduction due to space weathering, credits: 10.1038/s41550-022-01841-6.

Developing new catalysts for green hydrogen peroxide production

Physical Chemistry – Catalysis – Chemistry

Hydrogen peroxide (H_2O_2) is one of the top 100 most important chemicals globally, in high demand in both daily life and industrial processes, including disinfection, sanitisation, wastewater treatment, paper pulp bleaching, chemical synthesis and textile production. It is considered an excellent 'green' oxidant due to the absence of by-products (except water) upon use, relatively high redox potential, relative safety and low toxicity. However, the current industrial production method, the anthraquinone process, is energy- and waste-intensive, requiring complex infrastructures. Therefore, a more eco-friendly and sustainable alternative technology for hydrogen peroxide production is needed. Selective electrocatalytic oxygen reduction reaction (ORR) via the 2-electron pathway appears to be an attractive and feasible route that enables portable, on-demand, and distributed hydrogen peroxide synthesis. However, hydrogen peroxide production through ORR requires highly active and selective electrocatalysts.

Although platinum-group metals (PGMs) are known to be state-of-the-art ORR catalysts, their scarceness and low mass activity significantly hinder their practical use, calling for alternative electrocatalysts. Single-atom catalysts (SACs) have the potential for catalysing the ORR. However, they suffer from limited activity and selectivity, and we currently lack methods to improve their performance. Therefore, it is important to develop synthetic strategies to obtain SACs with tuned coordination environments and electronic structures that can enhance catalytic performance for realising highly efficient hydrogen peroxide electrosynthesis.

A team of researchers from China performed Annular Dark-Field Scanning TEM (ADF-STEM) at ePSIC as part of a project to develop a highly selective and active Co-N-C electrocatalyst for hydrogen peroxide electrosynthesis. The setup at ePSIC enabled them to minimise the sample damage caused by the electron beam and identify the metal atoms dispersed on the graphene support as bright dots.

The team successfully developed a facile and transient microwave irradiation treatment to simultaneously achieve the regulation of the coordination number and the surrounding oxygenated functional groups in cobalt-nitrogen-carbon SACs. The as-prepared catalyst possesses a low-coordinated Co-N_2 configuration and high content of C-O-C epoxide groups (Co-N_2-C/HO). Compared to the conventional Co-based SAC, Co-N_2-C/HO shows a significantly enhanced performance for hydrogen peroxide production with a high selectivity, prominent mass activity and large kinetic current density, making it one of the most active SACs for hydrogen peroxide electrosynthesis.

Considering the generality of the present synthetic methodology, this work offers a pathway toward the exploration of catalysts with unconventional structure and composition for catalysing reactions beyond ORR, such as CO_2 reduction and N_2 reduction reactions.

Related publication:
Gong, H. *et al*. Low-coordinated Co-N-C on oxygenated graphene for efficient electrocatalytic H_2O_2 production. *Advanced Functional Materials* **32**, 2106886 (2022). DOI: 10.1002/adfm.202106886

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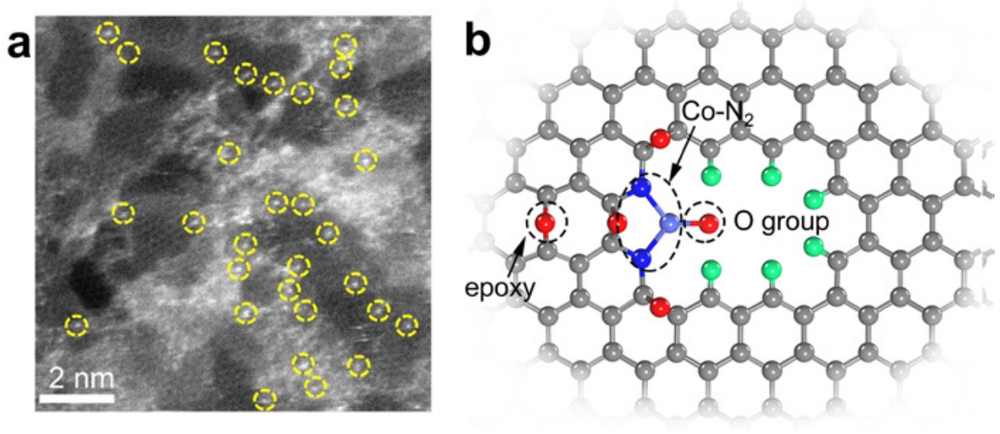

(a) ADF-STEM image of the graphene supported Co-N_2-C/HO catalyst, revealing that the Co metals are dispersed as individual atoms highlighted by the yellow circles. (b) Scheme of the structural model for the Co-N_2-C/HO catalyst, where the Co atom is coordinated to two N atoms and one O atom and the graphene substrate is decorated with epoxy groups. The green, grey, dark blue, red, and blue spheres represent the H, C, N, O, Co atom, respectively.

Introducing DIAD, Diamond's new Dual Imaging And Diffraction beamline

Materials – Technique Development

Diamond's newest fully operational beamline is DIAD, the Dual Imaging and Diffraction (DIAD) beamline. DIAD was born from a scientific need to simultaneously obtain the 3D microstructure of a material and local information about its phase composition and strain. DIAD's unique dual-beam setup is the world's first beamline that can quasi-simultaneously perform X-ray imaging and diffraction.

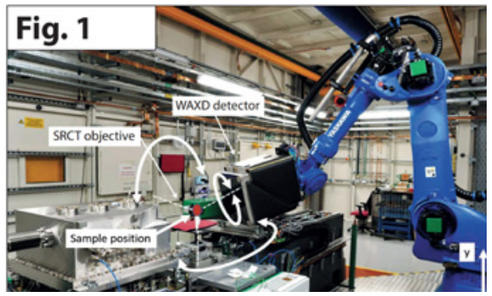

Fig1: Correlative SRCT and WAXD imaging at DIAD. (a) Annotated photograph showing moveable WAXD detector mounted on robot arm, and static SRCT objective.

The unique set up offers full/field imaging and tomography of 2D and 3D complex structures. Diffraction data is collected by a multi-axis robot arm mounted with a state-of-the-art Cd-Te Pilatus 3M detector (Fig 1). Users can perform spatially resolved phase identification; perform strain mapping using micro-diffraction (Fig 2); and perform in-situ and operando experiments that require spatially correlated results. Importantly, the simultaneous acquisition of imaging and diffraction data reduces lost down-time to mode switching, that is typical of other imaging and diffraction beamlines.

Experimental workflows on DIAD, post-processing, and analysis pipelines can all be automated using innovative in-house software. Users can access advanced data acquisition strategies, analyse their data live, and ultimately make informed decisions about their science to maximise the potential of their experimental time.

DIAD saw its First Light in December 2018 and welcomed its first User in February 2021. Since then, it has hosted a wide range of users across several fields including, but not limited to:

- **Medicine** - The behaviour of arterial stents under changing pressure for better patient outcomes
- **Environment**: The dynamic study of plant root behaviour in climate change affected soils
- **Energy** – The operando spatial distribution of charge in Li-ion batteries.
- **Materials** – Stress induced cracking in nuclear containment materials.

In summary, users can simultaneously collect time- and spatially-resolved information on both the crystallographic micro-structure and material macro-structure. The DIAD beamline has the unrivalled capability to access multi-scale, multi-modal information, granting users the ability to investigate highly dynamic systems.

Related publication:
Reinhard, C. *et al.* Beamline K11 DIAD: A new instrument for dual imaging and diffraction at Diamond Light Source. *Journal of Synchrotron Radiation* **28**.6: 1985-1995 (2021). DOI: 10.1107/S1600577521009875

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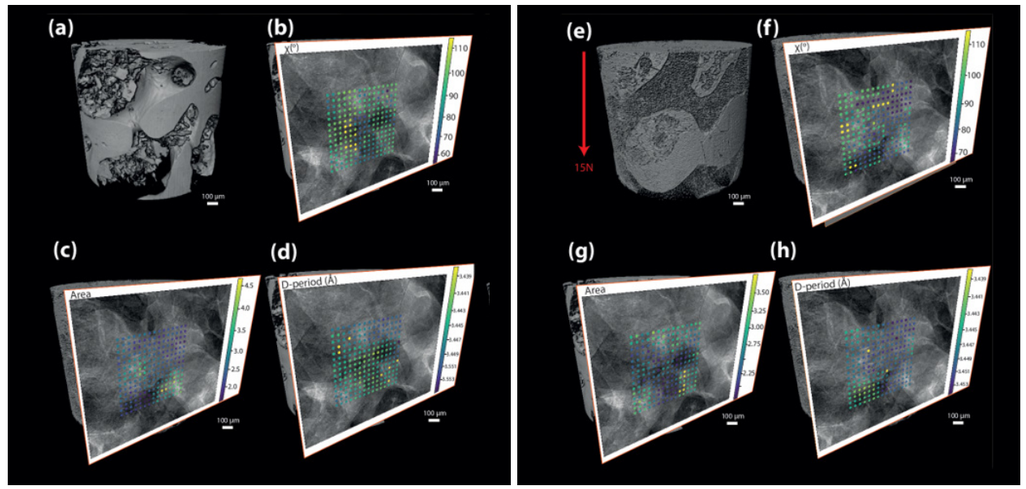

Fig2: 3D mapping of microscale and nanoscale compressive strain in Bovine tissue. Comparison of samples of unloaded (left grid) and 15 N compressive load (right grid). Figure highlights the multimodal capability of combined diffraction (raster-scanning mode) and tomography. Microscale structure (a,e); orientational texture (b,f); integrated 004 peak intensity (c,g), and lattice spacing (d,h). Created in Avizo (Source - MG27983 E. Newham, H. Gupta & J. Tozzi).

Crystallography Group

Joe Hriljac, Science Group Leader

The Crystallography Group comprises the High-Resolution Powder Diffraction beamline (I11), the Extreme Conditions beamline (I15), the X-ray Pair Distribution Function (XPDF) beamline (I15-1), and the Small-Molecule Single-Crystal Diffraction beamline (I19). The staff at the beamlines are not only expert in supporting research at their own beamlines but most have broader knowledge of the other beamlines and available techniques. This allows the group to fully exploit the technical and scientific capabilities to support research over a broad range of topics that include biomaterials, catalysis, energy production and storage, environmental understanding and remediation, and fundamental physics and chemistry. Studies under *in situ* and *operando* conditions are common to further enhance the breadth of scientific studies. Major upgrades to I11 and I15-1 started in past years are nearing completion and these will improve the quality of data that can be collected as well as provide greater automation and capabilities for user operations. Operations at I15 and I15-1 have been hampered by the need to upgrade the superconducting wiggler, but this is now back to routine operation and the user programs are resuming.

I11 update

The high brightness beamline uses monochromatic X-rays in the range of 6 - 25 keV for high-resolution and time-resolved powder diffraction experiments in the first Experimental Hutch (EH1) or for Long Duration Experiments in EH2. The varied science program supports a wide range of studies by chemists, physicists, materials scientists and environmental scientists in particular for non-ambient applications and experiments requiring unusual hardware setups such as toxic/corrosive gas absorption studies at cryogenic temperatures, resonant diffraction at high temperature and time-resolved in operando lithium-ion (Li-ion) battery work.

After running for over ten years, many components such as the monochromator, diffractometer and Multi-analyser Crystal (MAC) detector began to show signs of wear. An upgrade plan, endorsed by the Scientific Advisory Committee (SAC) and the Diamond Industrial Science Committee (DISCo) at the end of 2017 to replace these components started in 2019 and the new Newport diffractometer was partly installed when the site was shut in March 2020. During the course of the remainder of 2020 the installation and commissioning resumed when possible under the Covid-19 working protocols and finally in January 2021 the last stage, commissioning of the robot sample changer, was completed and the beamline became operational again. Since that point a large number of more routine experiments have been conducted by remote user access with beamline staff assistance and, more recently, users have returned to site. The construction of a new PSD using Mythen3 technology is well underway with hopes for completion, testing and commissioning in summer of 2023. This will lead to the ability for even faster collection of powder patterns for, *e.g.*, time-resolved studies.

I15 update

The Extreme Conditions beamline, I15, employs high energy X-rays to explore the structure of materials at high pressures, high and low temperatures, as well as other in situ and in operando conditions. The beamline receives an X-ray continuum from the superconducting wiggler; this allows for experiments that require monochromatic X-rays between 20 and 80 keV. I15 was originally designed to serve the mineral physics community, which it has, whilst also assisting material scientists, chemists and solid-state physicists with their structural investigations, at pressure or otherwise.

I15 continues to offer extensive capabilities and support to users to assist their high-pressure studies. I15 users have pre-experiment access to bespoke assistance and training from our highly skilled staff in diamond anvil cell (DAC) preparation and loading, as well as the usage of beamline DACs for novice users for I15 experiments. The high-pressure gas loader available at I15 offers users the choice of many possible gases to use as their pressure transmitting media (PTM), allowing them to optimise for hydrostaticity with helium or neon, or choosing a PTM based on desired interactions with the sample at pressure. The recent addition of the laser heating and resistive heating systems adds further capability – the I15 laser system is capable of quickly ramping the laser power to perturb a sample without delivering too much heat to the bulk. A DECTRIS PILATUS3 X CdTe 2M is now in routine operation and it provides much greater sensitivity to high-energy X-rays and the capability for much faster data collections. The quality of data has led to a resumption of the development of high-pressure single crystal data collection including the project of a Diamond PhD student. Further upgrades to I15 to take full advantage of fast hardware-based scanning and mapping are planned.

I15-1 update

The XPDF beamline, I15-1, is dedicated to producing high-quality X-ray scattering data for Pair Distribution Function (PDF) analysis. Operational since 2017, I15-1 has illuminated samples from diverse fields, from Earth sciences to pharmaceuticals, as well as material science and chemistry. XPDF receives X-rays from the inside edge of the wiggler fan, and this light is monochromated and directed to the end station in three energies: 40, 65 and 76 keV. PDF data are collected at high energies to produce the low sample absorption and high Q-range required for successful interpretation. Gaining structural information on amorphous samples is a primary goal of many XPDF experiments, but crystalline samples can also display local structure variations such as defects and disorder, which can be studied via PDF analysis. PDF data collections are rarely available at home institutions, so in order to allow more people to exploit this powerful technique I15-1 complements the standard proposal route with popular Rapid and Easy Access routes, where PDF data can be collected via a mail-in procedure.

Consisting of a sample position, with an optional sample-changing magazine, and two large area detectors, the end station is highly flexible and has been adapted to many in situ and in operando experiments, including variable temperature, gas flow, hydrothermal synthesis and electrochemical cycling. For more routine measurements, a sample-changing robot with 400 positions is used and was part of the SAC approved upgrade project. The final aspect of the upgrade is a bespoke new detector designed and constructed by the in-house Detector Group. It is based on CdTe sensors that will be much more sensitive at high energy and with faster electronics for data readout. Construction has progressed and the detector is currently in the testing stage with an expectation that it will be commissioned on the beamline in 2023. These upgrades will be a synergistic addition to the existing autoprocessing infrastructure and will allow users to collect better data with less manual intervention.

The Crystallography Science Group members.

> *“The staff at the beamlines are not only expert in supporting research at their own beamlines but most have broader knowledge of the other beamlines and available techniques.”*

I19 update

The Small-Molecule Single-Crystal Diffraction beamline, I19, uses X-rays in the 5 – 25 keV energy range to determine the structures of small-molecule and extended three-dimensional systems, *e.g.* Metal-Organic Frameworks, with single-crystal diffraction techniques. These methods can be applied to the characterisation of novel materials or for investigating the variation in the structure of a crystalline material under an external physical influence such as a change in temperature, the exposure to a gas, photo-excitation or through the application of high-pressure.

The use of the robotic sample changer, and remote access, is now well established in Experimental Hutch 1 (EH1) of the beamline, where premounted samples are sent to Diamond under cryogenic storage, and users then run their beamtime from their home institutions. This mode of operation makes it possible to carry out chemical crystallography studies in a more responsive manner as beamtime can be scheduled in more regular, and shorter, periods. We now schedule individual shifts, rather than whole one-day (three shifts) blocks of beamtime, for those wishing to run their beamtime via the remote access route. For Experimental Hutch 2 (EH2), we have recently developed a cell which allows a high static electric field to be applied to the sample crystal. The application of electric fields to materials can result in a variety of responses that may have important technological applications, spanning electronic and ionic conductivity to piezo- and ferro-electricity. Upgrades to the original mirrors was made in 2021 giving much improved performance in both beam focus and positional stability. During 2021 a DECTRIS EIGER2 X CdTe 4M was installed in EH2 and this is now in regular use producing vastly superior data over the older system.

Designing 'smart' crystalline membranes for graded molecular sieving

Surfaces - Physics - Chemistry - Materials Science - Interfaces and Thin Films - Organic Chemistry

Membrane technology provides a promising means of separating molecules as it offers greater selectivity than energy-intensive methods like distillation and chromatography. However, achieving the ideal structure and porosity to separate molecules of similar size remains a significant challenge.

A team of researchers from the University of Liverpool and Imperial College London aimed to fabricate a crystalline membrane using a porous organic cage molecule (POC). It has been reported that POCs are molecules with cavities that can create porosity in porous liquids, molecular crystals and amorphous solids, and host guest molecules. The researchers hoped the guest-accessible cavity would facilitate selective diffusion through the membrane structure. POCs can also undergo exciting structural transformations in crystalline solids in response to chemical stimuli.

The first step in their investigation was proving the membrane was crystalline. They then needed to investigate the membrane's dynamic behaviour to determine how it behaved in different experimental conditions, such as during filtration experiments.

They used Diamond's I11 beamline to perform Powder X-ray Diffraction studies and I07 beamline for Grazing Incidence X-ray Diffraction studies. The data collected at Diamond enabled them to determine the structure and study the dynamic behaviour of the crystalline membrane during *in situ* measurements. A vital aspect of the study is underpinned by the dynamic behaviour of the membrane and its ability to switch its pore aperture during filtration experiments in response to different chemical environments. At Diamond, the scientists could replicate the conditions used in larger-scale separation processes and study the structure and dynamic behaviour of the membrane.

The study found a highly ordered crystalline membrane with a switchable phase transition between two crystalline forms with different pore apertures. Both forms showed excellent separation performances. By varying the water/methanol ratio, the film can be switched between the two phases with different selectivities, giving a single, 'smart' crystalline membrane that can perform graded molecular sieving. The team used the dynamic behaviour of the membrane to perform graded molecular sieving experiments to separate a mixture of three organic molecules using a single, smart membrane.

Smart membranes that perform graded molecular sieving experiments to separate complex mixtures of molecules would create a parallel technology to the widespread and highly effective use of solvent gradients in chromatography. At the same time, membranes with switchable pore apertures could also lead to new applications in triggered drug delivery, biosensors, or fermentation/fractionation processes. Although the present method of synthesis poses a challenge to the scalability and implementation of POC membranes in commercial processes, there is potential for the development of a more scalable production method by utilising the solution processability of these molecular cages.

In the future, computational techniques, such as crystal structure prediction, will be employed to design POC crystals with desired properties based on first principles.

Related publication:
He, A. *et al.* A smart and responsive crystalline porous organic cage membrane with switchable pore apertures for graded molecular sieving. *Nature materials* **21**, 463–470 (2022). DOI: 10.1038/s41563-021-01168-z

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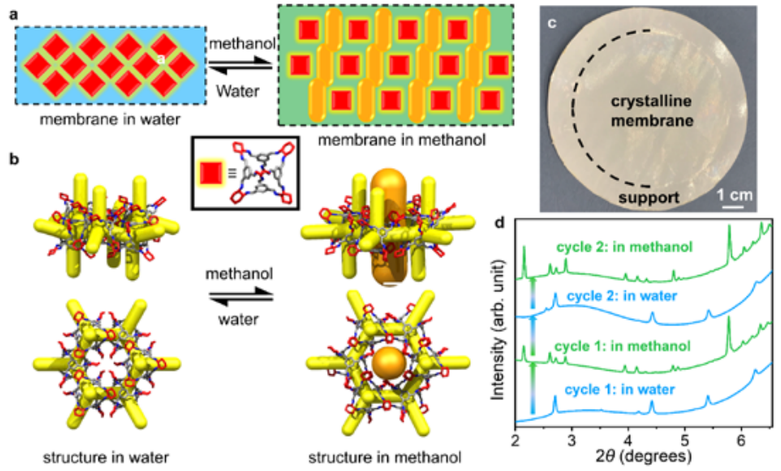

a) Graphical representation of membrane structure in water (blue) and methanol (green). b) Crystal structure with its 3-D pore network shown in yellow, found in water. A second structure was formed by soaking the membrane in methanol, which has additional extrinsic solvent-filled channels, shown here in orange, that open up additional porosity in the membrane in response to the methanol solvent. c) Photograph of the crystalline membrane with a diameter of 7.4 cm deposited on polyacrylonitrile support. d) In situ diffraction patterns of the crystalline membrane showing the reversible phase transition between the water and methanol structures.

Porous framework materials compress like a spring under high mechanical pressure.

Chemistry - Materials Science - Metal-Organic Frameworks - Coordination Chemistry - Solid-State Chemistry

Metal-organic frameworks (MOFs) are modular porous materials possessing a wide range of functions. Some MOFs reversibly switch between two different states, an open pore state with voids accessible for guest molecules and a closed pore state, where the voids of the framework are inaccessible for guest molecules. Usually, this transition is discontinuous, *i.e.* the material behaves like a switch, and only two different states/structures are accessible.

Researchers from Technische Universität Dortmund, the Technical University of Munich and the University of Edinburgh wanted to generate a MOF system featuring a continuum of available states/structures between the open and the closed states.

Hydrostatic mechanical pressure is a suitable stimulus to probe the structural response of MOFs. They investigated the high-pressure structural behaviour of a series of MOFs of the ZIF-62 family, which feature the same framework structure but possess various fractions of small- and large-sized organic linker molecules. They studied the influence of the molar fraction of the larger organic linker on the relative stabilities of the open pore and the closed pore states.

They used Diamond's I15 beamline because it is ideally suited for High-Pressure Powder X-ray Diffraction experiments, the method of choice to study the behaviour of crystalline materials under hydrostatic pressure. At I15, they could use a dedicated hydraulic high-pressure cell for the experiments. The hydraulic cell is designed for studying materials under pressure in the range from ambient up to 4000 bar, which is ideal for studying soft MOFs. In addition, the high quality of the data collected during the experiment meant that atomist structural refinements were possible.

The researchers found that all the studied ZIF-62 MOFs reversibly switch between an open pore and a closed pore state when a certain threshold pressure is reached. The required pressure for pore closure increases with increasing fraction of the large-sized linker included in ZIF-62. For very large fractions of the larger linker, ZIF-62 continuously transforms from the open pore to the closed pore form with increasing pressure. The structural change is similar to an inward folding of the network structure with increasing pressure. Structure refinement and detailed analyses revealed that the pore size of the continuously transforming ZIF-62 derivative also gets continuously narrower and narrower with increasing pressure.

The pressure driven open-pore to closed-pore transition of ZIF-62 could lead to applications of these materials as shock absorbers or nano-dampers. Moreover, the continuously changing pore size of the ZIF-62 derivative with a large fraction of the bulkier linker could be used to design pressure-switchable gas separation membranes. The material can be fine-tuned for a specific molecular separation task by applying a pressure that sets an appropriate pore size cut-off.

Related publication:
Song, J. *et al.* Tuning the high-pressure phase behaviour of highly compressible zeolitic imidazolate frameworks: from discontinuous to continuous pore closure by linker substitution. *Angewandte Chemie International Edition,* (2022). DOI: 10.1002/anie.202117565

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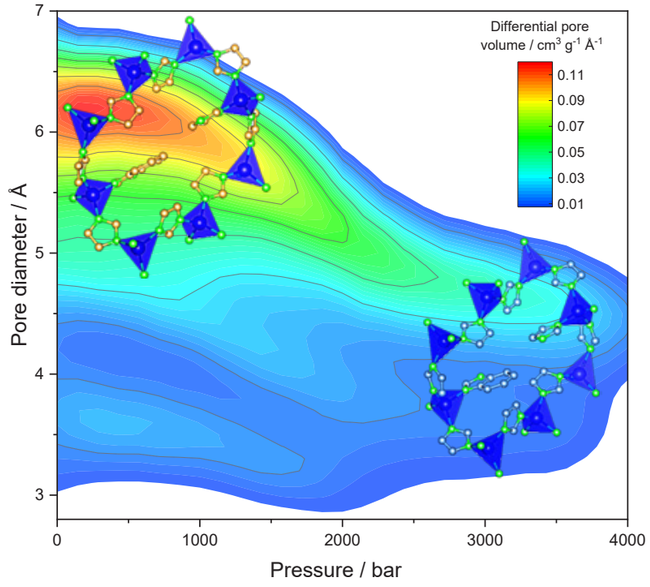

Variation of the pore size distribution of ZIF-62 as a function of hydrostatic mechanical pressure visualised as colour map. Representative 8-ring fragments of the crystal structures of ZIF-62 at 1 bar (left) and 4000 bar (right) are shown as insets. Zinc atoms are dark blue, nitrogen atoms are green and carbon atoms are orange / light blue. Hydrogen atoms are not shown.

Local cation ordering in 'disordered' Li-ion cathodes

Energy Storage - Energy - Physical Chemistry - Energy Materials - Chemistry - Materials Science

The modern world relies on high-performance lithium-ion (Li-ion) batteries to power mobile devices and electric-powered vehicles, and for the storage systems needed to ensure continuous supplies of low-carbon energy. Demand for these batteries is increasing, but current cathode materials limit the energy density and dominate the cost. In recent years, there has been a surge of interest in lithium-rich cathodes with a cation Disordered Rock Salt (DRS) structure. DRS cathodes are relatively low cost, and their high first charge capacities offer tantalising promise for high-energy-density Li-ion batteries with capacities vs Li/Li^+ beyond 300 mAh g^{-1}. However, DRS cathodes suffer from a large irreversible capacity loss on the first charge-discharge cycle.

The charge compensation mechanism responsible for the large first charge capacity and the origin of the capacity loss in DRS are not well understood. Understanding these phenomena requires *operando* investigations of structural and charge effects. Researchers therefore used *operando* techniques to investigate the evolution of the average structure, short-range ordering and charge during the electrochemical cycling of a DRS, using both spectroscopic and structural probes. To understand the structural evolution of nanostructured DRS cathodes *operando*, they acquired pair distribution function (PDF) data at Diamond's I15-1 beamline using Diamond Radial In Situ X-ray (DRIX) electrochemical cells. Performing *operando* High Energy Resolution Fluorescence Detected (HERFD-)XANES measurements at the I20 beamline provided a better understanding of the charge compensation mechanism.

The combined use of advanced PDF refinement methods and Bond Valence Sum (BVS) mismatch mapping revealed the local cation ordering of Li-ions with battery cycling can perturb the percolating Li-diffusion network in DRS. Total scattering and X-ray Emission Spectroscopy (XES) show that the cation and lithium vacancies in the layered domain that form during cycling become less accessible in subsequent charge cycles. The trapping of Li-ions in short-range-ordered domains could be associated with the capacity fade of DRS and could be a significant source of capacity fade alongside contributions from oxygen redox irreversibility.

The key to reducing capacity losses of DRS could be in preventing the formation of layered domains during cycling or controlling their size. Potentially, this could be achieved via nanostructuration or by introducing electrochemically inactive dopants. Future research should investigate the formation of short-range ordering in DRS cathodes, whether it forms during ball milling used to prepare the composite or through evolution of these domains with electrochemical cycling. Although the growth of layered domains can act as a trap for lithium, layered cathode materials have an inherently high capacity and rate capability. Further investigation into the interplay between the layered and DRS sublattices is needed, and the impact of short-range ordering should be optimised.

This work provides insight into the design of better DRS cathodes and highlights the importance of local structures in the cyclability of battery materials. Furthermore, successful control of the coexistence of layered and DRS sublattices offers a novel route to electrode design, opening a new path to developing high-performance cathode materials. This research is an example of how multimodal, *operando* experiments across complementary techniques including HERFD-XANES, XES (Kβ main line, V2C), and X-ray Total Scattering (Bragg, XPDF), can aid the complete understanding of complex electrochemical processes in new battery materials.

Related publication:
Diaz-Lopez, M. *et al.* Li trapping in nanolayers of cation 'disordered' rock salt cathodes. *Journal of Materials Chemistry A* **10**, 17415-17423 (2022). DOI: 10.1039/D2TA04262B

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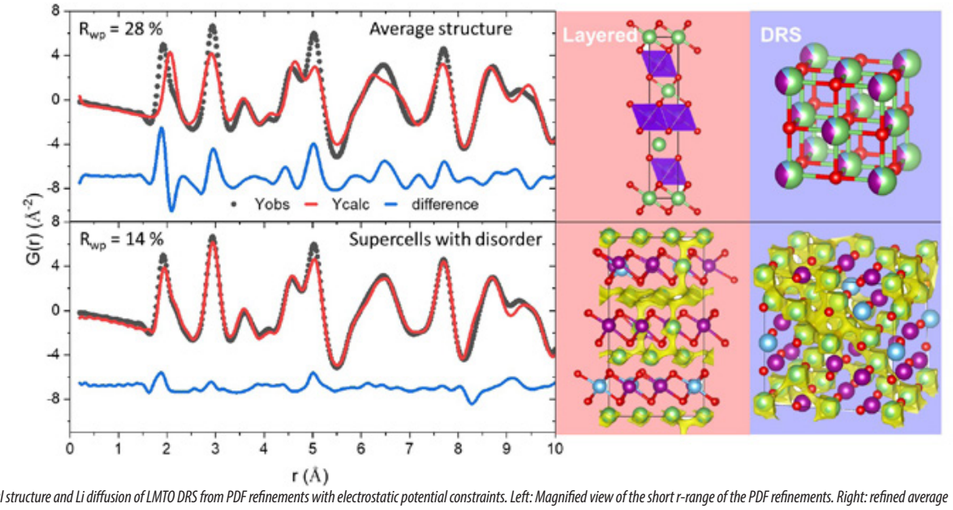

Local structure and Li diffusion of LMTO DRS from PDF refinements with electrostatic potential constraints. Left: Magnified view of the short r-range of the PDF refinements. Right: refined average structural models (top) and supercells of atomistic disorder (bottom). Green, purple, blue and red spheres denote Li, Mn, Ti and O, respectively. The yellow isosurfaces represent the regions with an energy threshold suitable for Li-diffusion. This figure is taken from our published work (https://doi.org/10.1039/d2ta04262b).

Electron movement between components of mechanically interlocked molecules

Physical Chemistry - Chemistry

Controlling photoinduced charge transfer within molecules is a significant challenge. An international team of researchers previously demonstrated that a macrocycle (wheel-shaped molecule) could reversibly lose an electron. They wanted to see if threading an electron-accepting molecule through this macrocycle could make a mechanically interlocked system, known as a rotaxane, where the electron could be stimulated to hop from the wheel to the thread. They made two rotaxanes with different numbers, types and positions of macrocycles to compare electron movement in these different systems.

In order to track the location of electrons before and after excitation, they needed to investigate the optical properties of the rotaxanes in different oxidation states and then perform time-resolved spectroscopic studies after photoexcitation of the molecules to determine the pathways of electron transfer in the molecule. To appreciate all these factors, it was vital to prove the orientation and arrangement of the complex structure of the threaded molecule. Although the structure could be evidenced by NMR spectroscopy, obtaining a crystal structure provided a more elegant and accurate determination of the molecular arrangement.

They obtained the single crystal structure of the most complex compound, a hetero[4]rotaxane, using data from Diamond's I19 beamline. A [4]rotaxane is a molecular thread passing through three wheel-shaped molecules. Crystal structures of such intricate molecular systems are incredibly rare, so it was valuable for this work to obtain this structure that showed the position and arrangement of all the interlocked pieces.

Crystals of this compound are both small and very weakly diffracting. Much of the team's research focuses on crystal engineering – fine-tuning properties and positions of active groups within a crystal. For this, good-quality crystallography data is essential. Diamond, particularly beamline I19, can provide high-energy X-rays for crystallography studies, allowing scientists to investigate crystals smaller than those they can study in their labs. This is particularly pertinent for challenging molecules, such as mechanically interlocked molecules, which often form small or weakly diffracting crystals. The intensity of the X-ray source at Diamond enables the collection of more accurate information about the arrangement of atoms within the target molecules.

In this study, combining the data from the synchrotron X-ray source with electron diffraction studies allowed the complete determination of the structure of this fascinating molecule.

The movement of the electron can be stimulated with photons (laser light), which causes an electron to rapidly hop from the wheel to the thread. This event occurs at different rates in the different systems, which led the research team to conclude that a distinct wheel molecule was supplying the electron in each system.

Understanding how to control the movement of electrons in complex molecular systems is useful in the construction of organic electronic devices, such as OLEDs and solar cells. This study demonstrates the use of a relatively compact electron-donating molecule that can easily be incorporated into more complex molecules. Exploring a mechanically interlocked system, specifically a rotaxane, demonstrates how we might control the movement of electrons in a molecular machine.

Related publication:
Pearce, N. *et al.* Selective photoinduced charge separation in perylenediimide-pillar[5]arene rotaxanes. Nature Communications **13**, 415 (2022). DOI: 10.1038/s41467-022-28022-3

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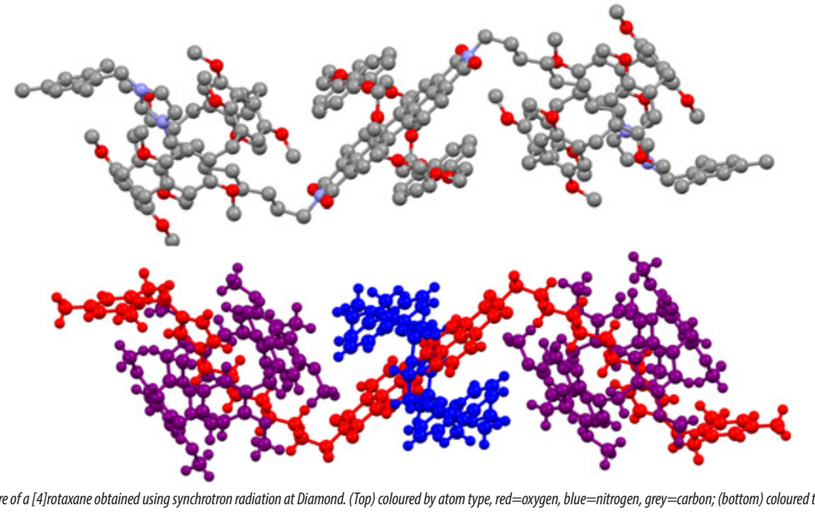


X-ray crystal structure of a [4]rotaxane obtained using synchrotron radiation at Diamond. (Top) coloured by atom type, red=oxygen, blue=nitrogen, grey=carbon; (bottom) coloured to show the distinct molecular components, red = thread, purple = wheels 1&3, blue = wheel 2.

Spectroscopy Group

Sofia Diaz-Moreno, Science Group Leader

The Diamond Spectroscopy Group consists of four beamlines; the Microfocus Spectroscopy beamline, I18, the Core EXAFS beamline, B18, and the two independently operating branches of the Versatile X-ray Absorption Spectroscopy beamline, I20-Scanning and I20-EDE. These four spectrometers are complementary in the energy ranges they cover, the size of their focussed beam spots delivered to the sample, and the time resolutions they are able to reach. This complementarity means that they can support research across many different scientific disciplines, from chemistry and catalysis through materials science, condensed matter physics, environmental and life science, energy materials and cultural heritage.

Many technical developments that enhance the capabilities of the group for *in situ* and *operando* experiments have been implemented on the spectroscopy beamlines over the past year. Some of the efforts have been focussed on upgrading the infrastructure of the beamlines. A Local Exhaust Ventilation (LEV) system has been recently installed on I18 to enable experiments with toxic and/or flammable gases, while the existing B18 LEV system has been upgraded, to remove the limitations that the previous system imposed on the sample environment that could be used. In addition, permanent gas monitors for the most commonly used toxic and flammable gases have been added to B18, linking them to the beamline alarm panel to increase the safety for associated experiments. A new sample environment has also been deployed on the beamlines. A new capillary reactor to perform gas experiments at high pressure, up to 20 bars, has been recently used for a user experiment on B18, and this system is now available for the other beamlines in the group. In addition, an OctoBoost16000 booster has been procured for the IVIUM OctoStats potentiostat, increasing the maximum current achievable from 1 A up to 128 A. This will enable the study of electrochemical systems at current densities comparable to the optimised cell designs used in practical applications.

I18 update

The Microfocus Spectroscopy Beamline, I18, uses a finely focussed beam down to 2 µm in size to investigate heterogeneous samples with a variety of experimental X-ray techniques, such as Fluorescence (XRF) and Diffraction (XRD) imaging, micro Absorption Spectroscopy (µXAS), and XRF and XRD micro-tomography (µCT).

In the past few years, improvements in the beamline hardware have allowed a significant speed-up in data collection, which is particularly beneficial for techniques that required long measurement times such as µCT-XRF and µCT-XRD. To keep up with these developments, this year has seen significant efforts invested in optimising the automatic data reduction and processing routines, so that processed XRF and XRD maps can now be shown on the fly, while tomography measurements are displayed only seconds after the scan is finished.

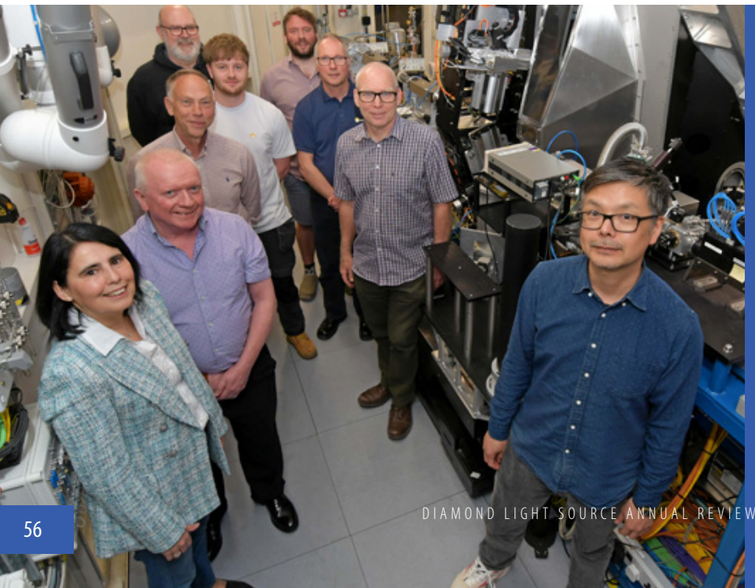
Transmission imaging capabilities have also been added to the plethora of techniques available in I18. This has been possible by the procurement of a new 5 Megapixel sCMOS 2D imaging camera that can record images at rates of up to 100 frames per second.

The design of the new in-house X-ray emission spectrometer is completed, and the procurement is now underway. Unfortunately delays with the delivery of some of the components have had an impact on the commissioning and installation schedules, but it is expected that the instrument will be available for the user community early next year. This instrument will expand the photon-in/photon-out capabilities of the group towards the tender energy regime, and the small focal spot of the beamline will enable spatially resolved measurements.

B18 update

The Core EXAFS beamline, B18, is optimised for the efficient collection of XAS data over all elements heavier than phosphorous. The focussing optics and the capability of the monochromator for continuous scanning, together with a flexible experimental space that supports a large range of sample environment equipment, make this beamline ideal to perform experiments under *in situ* and *operando* conditions.

Last year has seen a development in the fluorescence detection capabilities of the beamline. A new seven-element silicon drift detector has been procured to replace the four-element detector. The larger solid angle covered by the new detector, together with its faster pre-amplifiers, will reduce the collection time required to obtain the desired signal to noise ratios. In addition, a new Odin-based data acquisition software system has been developed for the 36-element Ge detector, allowing the XSPRESS-4 digital pulse processor readout system to be used to its full potential up to a data rate of 1 kHz. To facilitate experiments done in fluorescence detection mode, both fluorescence detectors have been motorised so that the sample to detector distance can now be optimised remotely, increasing the efficiency of beamtime usage.



Many technical developments that enhance the capabilities of the group for in situ and operando experiments have been implemented on the spectroscopy beamlines over the past year.

Figure 1. Science and engineering team who delivered the new I20 14-crystal spectrometer.

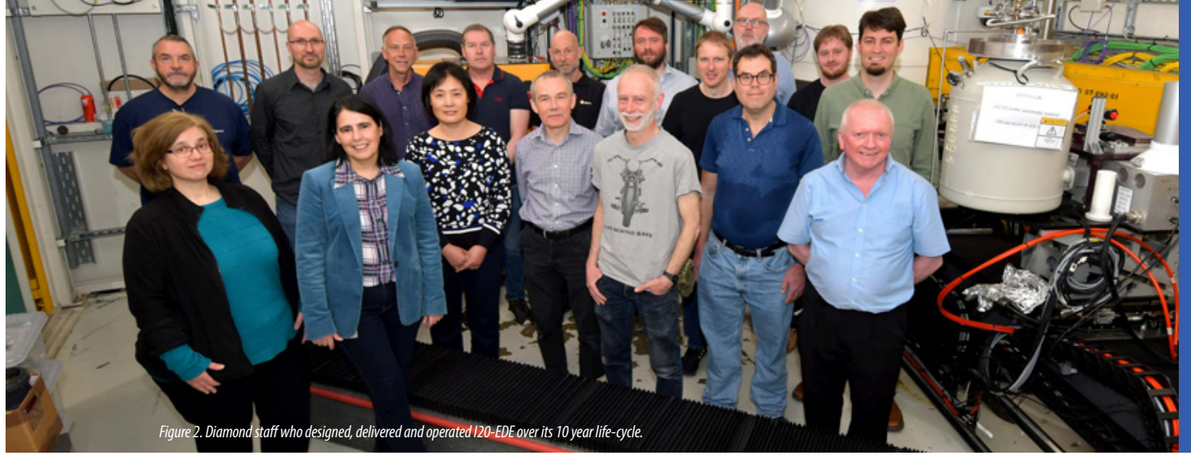


Figure 2. Diamond staff who designed, delivered and operated I20-EDE over its 10 year life-cycle.

To enhance the capabilities of the beamline in the tender X-ray regime, effort has been devoted to the development of new sample environment. A transfer system has been designed so air sensitive samples can be loaded directly from the glove box into the tender X-ray chamber. In addition, a new sample stick for the tender X-ray chamber has been integrated, allowing measurements of multiple samples down to 77 K.

To help users in the process of data acquisition and live analysis, an ISPyB webpage has been deployed for real time data reduction. Implementation of real time data analysis is in progress, including averaging of multiple repetitions and automatic outlier rejection.

I20-Scanning update

On I20-scanning, the high flux provided by the wiggler source is used in two end-stations. The X-ray emission end-station has been designed to perform high-energy-resolution fluorescence detection XAS (HERFD-XAS) and resonant and non-resonant X-ray emission spectroscopy (RXES and XES) studies, looking at the electronic state of materials. The XAS end-station is optimized for studying the structure of low concentration samples.

In October last year, the three-analyser crystal spectrometer used at the XES end-station was replaced by a fourteen-analyser crystal instrument (Figure 1). The beamline re-opened to users in January 2023, so several XAS experiments were performed while extensive cabling and commissioning of the spectrometer continued. The with-beam spectrometer commissioning took place last month, and HERFD-XAS, RXES and XES data were taken from reference samples. While the energy resolution achieved by the spectrometer is comparable to that of the old spectrometer, the intensity of the radiation collected is significantly higher. This will enable the study of more dilute systems, especially for experiments using Kβ emission lines, that are approximately one order of magnitude weaker than the Kα lines. The first user of the new spectrometer is scheduled for April 2023.

A project to develop a new four-bounce monochromator has begun; this will use liquid nitrogen direct cooling of the first crystal to maintain thermal stability and for the first time enable the beamline to utilise a Si(311) monochromator, reaching energies above 20 keV. The project will be undertaken in-house, capitalising on Diamond's expertise in building monochromators.

I20-EDE final update

The Energy Dispersive EXAFS (EDE) branch of I20 uses a polychromator to perform XAS experiments in a dispersive geometry. It is designed for *in situ* and *operando* studies with time resolutions ranging from seconds down to milliseconds or even microseconds.

EDE experiments are very specialised and, due to the unique dispersive setup, the sample quality requirements are much more stringent than for conventional XAS beamlines, as changes in background can compromise the normalisation procedure. This has meant that the I20-EDE branchline largely remained the tool of choice for research in a select number of scientific areas. A decision has been taken to close the I20-EDE beamline in April 2023 (Figure 2), so Diamond's limited resources can be deployed towards areas that are more in tune with user demand and anticipating the arrival of SWIFT in a few years (see below). The dedicated beamline team worked hard to maximise the beamline's output until the end of operations and there were thirteen experimental sessions in the final allocation period. In particular, the provision of an XRD detector on the beamline proved popular with users, giving them the possibility to interrogate their samples using two techniques simultaneously.

SWIFT update

The fast-scanning capabilities of the I20-EDE beamline will be covered by the new spectroscopy beamline that will be built as part of the Diamond-II upgrade programme. The new beamline, called SWIFT (Spectroscopy WithIn Fast Timescales) will be a wiggler-based, quick-scanning EXAFS beamline dedicated to *operando* studies, also at micrometric scale. SWIFT will become operational on Diamond-II immediately after the dark period that is currently expected to last for 18 months from December 2026.

SWIFT's design is progressing well. The layout, optics and specifications of the beamline are now defined, including the functionalities of the acquisition, controls and analysis software which will be based on the new Diamond-II stack. A preliminary engineering analysis of the Quick-EXAFS monochromator design has already been performed and the project is showing great promise.

Community Support and Development

As part of our on-going role to support the development of the spectroscopy user community, the Spectroscopy Group organized the annual three-day X-ray Absorption Spectroscopy workshop in June 2022. After running the workshop remotely the previous year, we were able to run it in-person, as one of the first on-site meetings organised at Diamond after the COVID pandemic. As in previous years, the workshop was in high demand, with more than 150 applications, although required staff to student ratios limited the successful applications to 32 participants. On this occasion, as well as a brief introduction to the spectroscopy beamlines at Diamond and the methods to process and analyse spectroscopy data, the workshop included a brief introduction to XANES modelling using DFT approaches.

The group is also invested in expanding further the capabilities for advance spectroscopy data analysis. With this aim, Diamond, though the Spectroscopy Group, is involved on the UK High-End Computing Consortium for X-ray Spectroscopy (HPC-CONEXS), an Engineering and Physical Sciences Research Council (EPSRC) funded High End Computing Consortium that will take the lead in the development and distribution of computational tools to advance the detailed analysis of experimental spectroscopy data. This consortium is an evolution of the very successful COllaborative NEtwork for X-ray Spectroscopy (CONEXS) project, started three years ago and is coming to an end at the end of April 2023.

Developing a deeper understanding of electrode materials for sodium-ion batteries

Energy Storage – Energy – Physical Chemistry – Energy Materials – Chemistry – Material Science – Nanoscience/Nanotechnology

Various materials can be used as electrodes in sodium-ion batteries (SIBs), and a comprehensive understanding of their charge storage mechanisms is essential for SIB development. Researchers from the Christian-Albrecht University of Kiel, in Germany, used X-ray Absorption Spectroscopy (XAS) on Diamond's B18 beamline as part of a rigorous study of the sodium storage properties of ultra-small Fe_3S_4 nanoparticles. This material exhibits excellent electrochemical performance as an anode material for SIBs.

Previous research using X-ray diffraction and Total Scattering (Pair Distribution Function analysis) had shown different structural phase transformations during the discharge and charge of the anode material. For a more detailed understanding, the researchers needed to analyse the local structure around the elements Fe and S and their oxidation states, in the pristine nanocrystalline material and during charging and discharging. Such element-specific information, combined with other techniques (*e.g.*, to determine the crystallographic structure), can yield important insights into the reaction mechanism of a battery material during operation and allow optimisation of battery cells, *e.g.*, by tailoring materials properties or by adjusting cut-off potentials.

B18 provides high-quality XAS data, which allow precise and reliable determination of changes at the K-edges and yield fantastic k space signal with high spectral resolution. During their experiments, the team gathered X-ray Absorption Near Edge Structure (XANES) spectra at the Fe and S K-edges (see figure) and Extended X-ray Absorption Fine Structure (EXAFS) spectra at the Fe K-edge. Their results showed that the Na storage mechanism of this anode material can be attributed to cationic redox chemistry involving Fe. The experiments revealed the oxidation states of both elements at specific discharge/charge voltages. Using this information in combination with findings from other techniques (multi-method analysis), the researchers were able to explain the long-term cycle stabilities of Na/Fe_3S_4 cells during cycling to different lower cut-off potentials.

XANES and EXAFS experiments yield insights into sodium storage mechanisms that are important for understanding and developing other electrode (anode and cathode) materials *ex situ* and for testing battery cells *in operando* during galvanostatic or potentiostatic measurements. The investigation of fundamental redox reactions in battery chemistry is a highly relevant topic to understand degradation reactions. Such studies can be used to find root causes for cell failure, precisely adjust battery cell limits and find optimal cycling conditions to improve the electrochemical performances and battery lifetime.

Related publication:
Hartmann, F. *et al.* Understanding sodium storage properties of ultra-small Fe_3S_4 nanoparticles - a combined XRD, PDF, XAS and electrokinetic study. *Nanoscale* **14,7**: 2696-2710 (2022). DOI:10.1039/D1NR06950K

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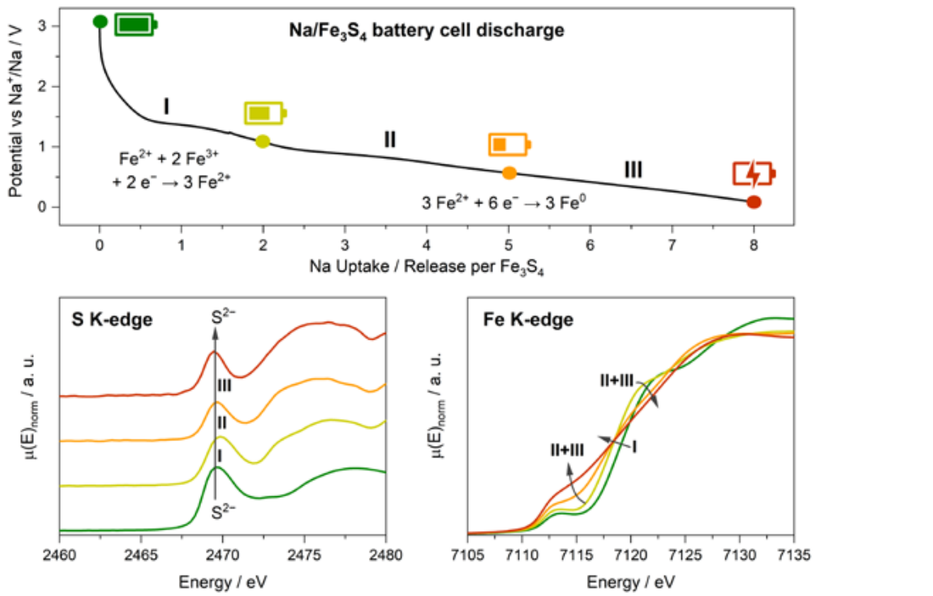

The first electrochemical discharge profile of the anode material nano-Fe_3S_4 vs Na^+/Na in a sodium-ion battery cell is divided into three steps (top). X-Ray Absorption Near Edge Structure (XANES) spectra at the Fe (bottom right) and S K-edges (bottom left) were obtained at Diamond Light Source (beamline B18) after uptake of certain Na amounts into the anode material and reveal the cationic redox chemistry during Na storage, which involves Fe^{3+}, Fe^{2+} and Fe^0 products, while the oxidation state of Sulphur remains -2.

Investigating fungus-plant-soil interactions using synchrotron techniques

Plant Science – Life Sciences & Biotech

Soil is the largest terrestrial carbon pool, larger than plant and atmospheric pools combined. However, we have very little understanding of what is happening in the soil. Because soil is opaque, it is challenging to unravel intricate plant-soil-microbiome relationships. An increased understanding of these relationships will have profound implications for current agricultural practices.

The soil microbiome, which includes fungi, is one of the largest compartments of the soil carbon pool. Phosphorus is essential for plant growth; however, mineral phosphorus resources are sparse and unevenly distributed across the world. Mycorrhizal fungi form symbiotic relationships with plants, acquiring phosphorus from distant sources and providing it to plants in return for carbon. Engaging in these mutualisms increases the total available nutrient pool for plants. However, little is known about how mycorrhizal fungi colonise soil pore-space, and models of phosphorus uptake enhanced by mycorrhizal fungi are poorly validated.

A team of researchers imaged soil and fungal structures in 3D and used X-ray Fluorescence Spectroscopy (XRF) coupled with X-ray Absorption Near Edge Structure (XANES) at beamline I18 to study the chemical impacts of the presence of mycorrhizal fungi in the soil. They also used the X-ray Computed Tomography (XCT) beamline at I13 to study how these plant-fungi relationships work in the soil, beyond what can be observed with the naked eye.

The XCT results helped them to understand where mycorrhizal fungi are predominantly present. Then they coupled this with XRF and XANES to understand the nature and impacts of mycorrhizal fungus and preferential uptake and mechanisms. Finally, they used advanced image analysis tools to correlate all imaging results and do further quantitative correlative analysis.

Using the analysis tool suite, they were able to i) uncover highly detailed subtleties in the preferential presence of mycorrhizal fungi in the soil previously not shown (*e.g.* organic matter, 'clay fraction', and soil mineralogy), and ii) put tight constraints on mycorrhizal fungus phosphorus uptake models. By employing correlative imaging, they were able to tighten this value down compared to previous macroscopic studies.

This study showed for the first time that it is possible to visualise mycorrhizal fungus networks in 3D within soil using synchrotron X-ray Computed Tomography, albeit as yet only in the soil pore-spaces.

In order to reduce the need to fertilise crops with phosphorus, there is a requirement to understand and establish alternatives for plant phosphorus acquisition. Symbiotic mycorrhizal fungi are an alternative as a soil treatment, but it is often unclear how much and how they can help with plant phosphorus uptake as all processes occur on very small (~1 micron) scales. Thus, it is important to do pore-scale structural and chemical imaging that underpins nutrient uptake and movement models. These models will then allow strategies for the most efficient use of mycorrhizal soil treatments to be found.

Related publication:
Keyes, S. *et al.* Multimodal correlative imaging and modelling of phosphorus uptake from soil by hyphae of mycorrhizal fungi. *New Phytologist* **234**, 688-703 (2022). DOI: 10.1111/nph.17980

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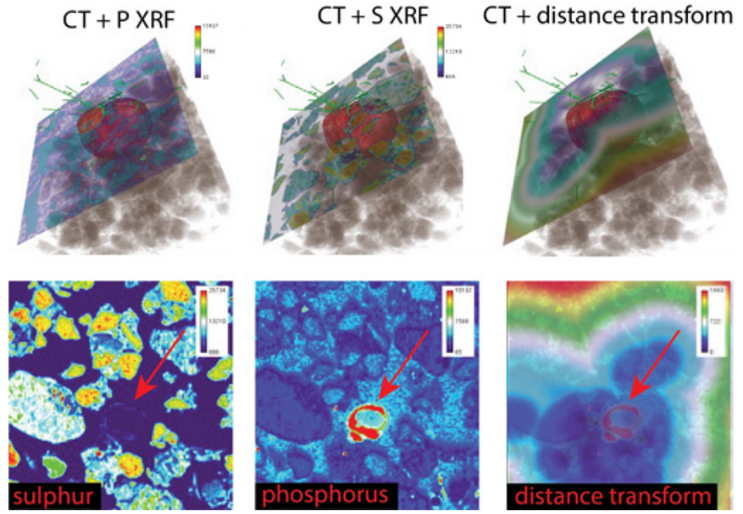

The combination of synchrotron X-ray Computed Tomography (SR-XCT) and synchrotron X-ray Fluorescence (SR-XRF) imaging. By utilizing both SR-XCT and SR-XRF, we were able to elucidate structural-functional relationships and intricacies for mycorrhizal fungi in soils in situ. The figure above is showing the preferential interaction of mycorrhizal hyphae with organic matter rich in both organic phosphorus and sulphur, identified via X-ray Absorption Near Edge Structure (XANES).

Investigating the damage tolerance of high-temperature superconductors for fusion power plants

High Energy & Particle Physics – Superconductors – Quantum Materials – Energy – Physics – Materials Science

High-temperature superconductors are an essential component of compact tokamak fusion reactors, which promise to provide a commercial route towards fusion power plants by the mid-2030s. However, there are many technical challenges with achieving this ambitious goal, including a lack of understanding of what will happen to the superconducting magnets bombarded with high-energy neutrons produced by the fusion reaction.

Researchers from the University of Oxford and Diamond Light Source sought to discover what kinds of defects are generated by irradiation with energetic particles in the high-temperature superconducting tapes used to make demonstrator fusion magnets by companies such as Tokamak Energy and Commonwealth Fusion Systems.

Superconductivity in high-temperature superconductors is strongly suppressed by structural disorder. When neutrons or other energetic particles collide with atoms in the superconductor, some get knocked out of position, creating defects in the crystal that reduce superconductivity. Figuring out what kinds of defects are produced during irradiation will help to determine, in combination with other experimental and modelling studies, how long these materials may survive under realistic operating conditions in a fusion reactor. That will inform engineering decisions, such as how much neutron shielding the magnets will require.

Using atomic resolution Electron Microscopy at ePSIC enabled the team to determine that the crystalline arrangement of the heavy metal ions in the structure remains largely intact, even at a dose where superconductivity has been completely lost. However, individual atomic defects are difficult to 'see' with an electron microscope, particularly the light oxygen atoms that turn out to be very important.

Using high energy resolution X-ray Spectroscopy, the I20-Scanning beamline allowed the team to probe changes in the local chemical bonding environment around the copper atoms that occur when oxygen atoms move to different sites in the structure.

By correlating with spectra simulated using first principles computational methods, the researchers identified specific crystal defects that may be responsible for the loss of superconductivity. Their results gave conclusive evidence for the first time that irradiation produces considerable changes to the environment surrounding the copper atoms that reside in the superconducting planes of the crystal. Prior to this, it had been speculated that oxygen atoms in the regions of the crystal between the superconducting planes would be more likely to be displaced.

Comparing the nature of the defects created in high-temperature superconductors under irradiation by different kinds of energetic species, and at different temperatures, is a key part of being able to predict how these materials will behave in operation in a real fusion device. Experiments with neutrons are very challenging and costly, so it is essential to identify suitable alternatives that can mimic neutron damage.

Diamond has recently opened a new Active Materials Laboratory, which has allowed the team to repeat their spectroscopy experiments on neutron-irradiated superconductors that are radioactive. This is a significant step towards qualifying which energetic particles produce the same type of damage as neutrons.

The fundamental understanding gained from these experiments will lead to more robust interpretation of a range of irradiation data. Ultimately, the hope is that it will provide magnet designers with more reliable information.

Related publication:
Nicholls, RJ. *et al.* Understanding irradiation damage in high-temperature superconductors for fusion reactors using high resolution X-ray absorption spectroscopy. *Communications Materials* **3**, 52 (2022). DOI: 10.1038/s43246-022-00272-0

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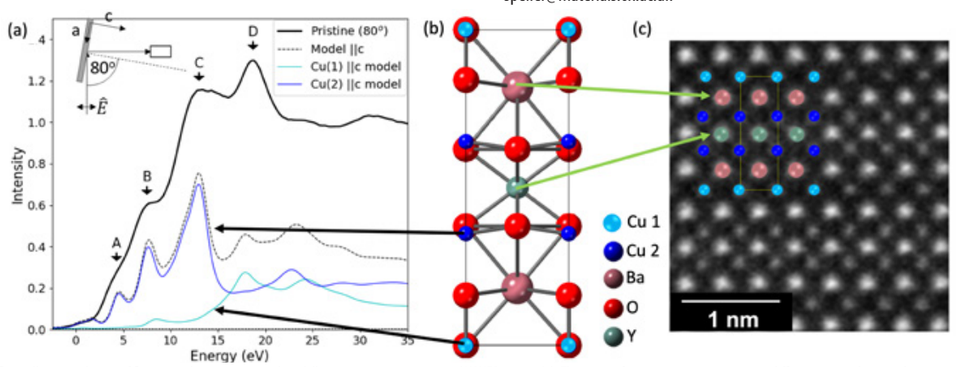

This figure illustrates the use of first principles computational modelling to interpret experimental XANES spectra (a). The spectra from copper atoms occupying different sites in the crystal structure, labelled Cu 1 and Cu 2 in (b), are calculated separately, enabling key features of the experimental spectra to be identified. Atomic resolution images, taken using the high angle annular dark field imaging mode in a scanning transmission electron microscope, show that the crystalline lattice of the heavy cations remains intact after irradiation (c). Data adapted from Commun Mater 5, 52 (2022) under Creative Commons Licence CC BY 4.0 (https://creativecommons.org/licenses/by/4.0/)

Control of zeolite microenvironment for biomass conversion

Bioenergy – Sustainable Energy Systems – Energy – Climate Change – Physical Chemistry – Catalysis - Chemistry

Pentadienes serve as key building blocks for the chemical and polymer industries and are widely used as monomers in the production of adhesives, plastics, and resins. However, state-of-the-art processes to produce pentadienes are based on steam cracking of naphtha (typically at 850°C) and rely on fossil fuels with the attendant environmental impacts. Therefore, the sustainable production of pentadienes from renewable resources, such as biomass-derived materials, is a vitally important and urgent task.

Methyltetrahydrofuran (2-MTHF) can be produced readily from lignocellulose-derived furfural via low-cost, high-yield processes and has been identified as a sustainable resource for making pentadienes via ring-opening, hydrogen transfer and dehydration processes. Leading catalysts for this reaction include amorphous SiO_2/Al_2O_3, and Al or B- zeolites. However, these microporous catalysts often suffer from deactivation due to the formation of cokes. Furthermore, achieving effective selectivity control towards pentadienes in this reaction is still a significant challenge.

MCM-41 is a mesoporous silica-based material used as a catalyst or catalyst support for a wide range of reactions; emerging niobium-based catalysts have shown exceptional performance for the hydrodeoxygenation of biomass under mild conditions.

An international team of researchers studied whether MCM-41 materials containing weak acid sites and active niobium sites effectively address the challenges of pentadiene production. The reaction mechanism of conversion of 2-MTHF is complex, involving multiple reaction intermediates and products. The ring-opening of 2-MTHF is the rate-limiting step in this conversion. The research team aimed to determine the full molecular details of the catalytic mechanism through the use of *operando* X-ray Absorption Spectroscopy (XAS), combined with Diffuse Reflectance Infrared Fourier Transform Spectroscopy (DRIFTS) and *in situ* high-field solid-state Nuclear Magnetic Resonance spectroscopy.

On Diamond's I20-EDE beamline, they used the spectroscopy group's recently commissioned high-temperature synchronous gas/vapour phase XAS/DRIFTS set-up coupled to the mass spectrometer and in-house developed gas dosing rig. This combination enabled them to propose a detailed reaction mechanism via temperature programmed spectroscopy.

This work reported the synthesis of a series of new (Al,Nb)-bimetallic mesoporous silica materials for the first time. AlNb-MCM-41(35/1/0.9) shows excellent catalytic performance for converting biomass-derived 2-MTHF to pentadienes.

The direct transformation of biomass derivatives to C5 dienes under mild conditions described in this study will have a significant impact on the development of future sustainable chemical processes. In addition, these findings have revealed the nature of Nb(V) sites during the conversion of 2-MTHF, and promoted the understanding of the catalytic role of Nb(V) sites in MCM-41, which will be of interest to those working in the fields of solid-state materials, catalysis and sustainable chemical production.

Related publication:
Fan, M. *et al.* Bimetallic Aluminum- and Niobium- doped MCM- 41 for efficient conversion of biomass- derived 2- Methyltetrahydrofuran to pentadienes. *Angewandte Chemie International Edition* ***51***,(2022). DOI: 10.1002/anie.202212164

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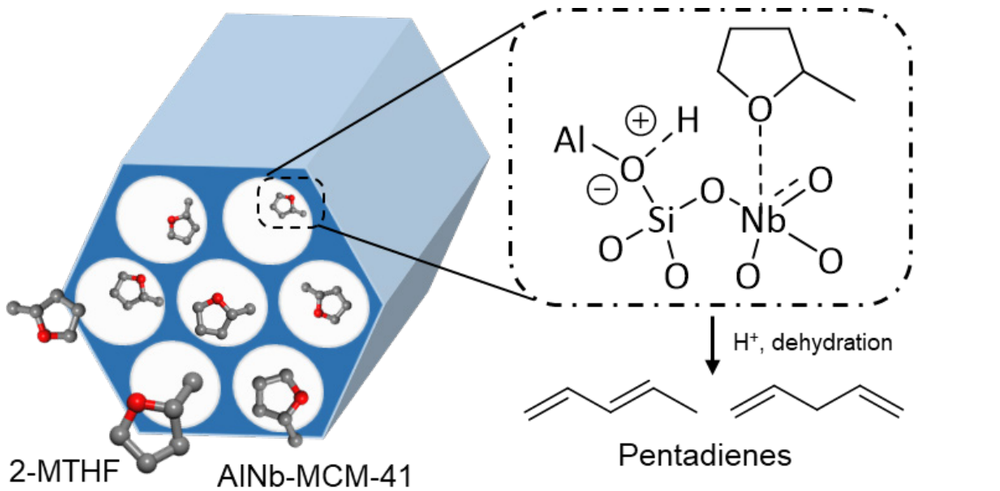

The highly selective conversion of biomass-derived 2-methyltetrahydrofuran (2-MTHF) into pentadienes has been achieved over an aluminium and niobium bimetallic atomically doped on MCM-41. The Nb(V) sites enhance the catalytic performance by binding 2-MTHF.

Soft Condensed Matter Group

Robert Rambo, Science Group Leader

The Soft Condensed Matter (SCM) Group is comprised of the High Throughput SAXS (B21), the Multimode Infrared Imaging and Microspectroscopy (MIRIAM) (B22), SAXS and Diffraction (I22) and the Circular Dichroism Microspectroscopy (B23) beamlines, co-located in zones 3 and 4 of Diamond. This unique portfolio of instruments enables studies of amorphous to noncrystalline materials at nano- to meso-scale resolutions that include two-dimensional thin-films (photovoltaics, OLEDs), living mammalian cells, three-dimensional matrices (e.g. metal-organic frameworks, gels and waxes) and micro/nano-particles (e.g. micro/nanoplastics) in any condensed matter states. SCM science is *"the science that underpins continued improvements to quality of life"* and was critical to the rapid development of the COVID vaccines from Pfizer-BioNTech and Moderna.

The SCM user community is international, nearly 70% of our peer-reviewed allocated beamtime were awarded to users from the United Kingdom where the remaining time is shared largely between member states of the European Union, United States, China, Canada, Japan, Israel, and Australia. In the last year, the SCM Group contributed to 132 scientific publications covering a broad range of disciplines including chemistry, material science, chemical engineering, physics and astronomy, biochemistry, genetics and molecular biology and engineering.

SCM group now provides off-line CD, InfraRed and SAXS measurement via peer-review applications and rapid access. The SCM Group maintains a dedicated laboratory space for visiting users. The laboratory houses vital equipment for sample preparation and analysis such as a centrifuge, a biosafety level 2 facility, spectroscopy equipment and the ability to work with different gases plus a cell culture lab for mammalian tissues (associated to B22). We acquired a new mass photometer by Refyn for single particle mass characterisations to complement our size-exclusion chromatography coupled, multi-angle light scattering (MALS) system (Wyatt and Agilent). In addition, we now house two incubators for supporting plant-based experiments. In collaboration with the DIAD beamline headed by Dr. Sharif Ahmed, SCM group will play a central role in building the food sciences and security user community at Diamond.

Diamond-II Machine Physics group has identified lattice dipole-quadrupoles (DQ) as suitable soft photon sources for B22 and B23. B22 shall see a significant increase in the available terahertz radiation with an ~1.3x increase in flux across the terahertz-to-infrared range producing one of the largest broad band sources spanning 3 decades in wavelengths for vibrational microscopy and imaging. Currently, we are investigating bespoke vacuum vessel designs for extracting the radiation and new front-end mirrors for directing the radiation through the existing port that separates the beamline from the synchrotron storage ring.

A new cohort of students joined our existing SCM doctoral students which now include the Universities of Pisa (Italy), Surrey and Chalmers, Southampton, King's College London, Imperial, Sheffield, Reading, Leeds Cambridge, and Durham. SCM provided several training workshops including the popular S4SAS meeting led by I22. B22 organised an advanced hyperspectral analysis and data processing training workshop with Soleil and Lubjiana University using machine learning software (Quasar) in IR image and spectroscopy analysis and B21 hosted a series of online, small group data analysis sessions for users which is now a routine part of our user program. In addition, we hosted the FoodBioSystems DTP partner event in October 2022. The event introduced Diamond's capabilities and resources to a new user community and included Aberystwyth University, Brunel University, Cranfield University, Queen's University Belfast, University of Reading and University of Surrey. The event attracted ~70 attendees and featured speakers from eBIC (Dr. Yuriy Chaban and Dr. Alisa Powell), I18 (Dr. Konstantin Igniatev) and DIAD (Dr. Sharif Ahmed).

B21 Update

B21 studies noncrystalline, randomly oriented particles using high-throughput approaches. SAXS measurements can be made on any type of sample and in any physical state. The life sciences community comprises our largest user group since such measurements provide the opportunity to study biological machines in conditions that are comparable to their liquid, hydrated environment. B21 started two major upgrades to be completed by 2023: 1) an automation project that will enable high-throughput studies of semi-solid materials and 2) WAXS detector upgrade. B21 pioneered mail-in SAXS services for UK and EU users. Prior to the COVID restrictions, mail-in experiments were limited to biological, liquid samples but was expanded to general soft condensed matter samples. The automation project will enable higher throughput of semi-solid materials using a 6-axis Meca500 robotic arm available through mail-in and peer-review proposals. The WAXS detector upgrade will be provided by an in-vacuum Eiger 1M detector. The increased, observed scattering range will further support SCM experiments on B21 whilst also adding additional capabilities to the sample environment unit (SEU). Currently, B21 experiments are performed at fixed temperatures (<60 °C), the new SEU will allow for routine measurements between (-4 and 120 °C), ideal for studying phase behaviour of proteins, waxes, gels and other polymer blends. In 2021, B21 commissioned a new SEU that enabled simultaneous UV-visible light illumination of the sample during X-ray exposure which has now produced its first paper examining time-resolved, photo-switchable polymers for solar fuels (Tyagi et al, JACS Au 2022, 2, 12, 2670-2677).

B22 Update

The Multimode Infrared Imaging and Microspectroscopy (MIRIAM) beamline, B22, is used to assess the molecular composition and microscopic spatial distribution of a sample at the highest, optically-achievable resolution in the infrared (IR). B22 operates two end-stations for scanning IR spectro-microscopy and IR imaging, with a suite of single and 2-D detectors that seamlessly cover the whole IR range, from near-IR to mid-IR and further into THz. B22 has been used in the analysis of inorganic-organic combinations in biomineralogy or composite materials, chemical degradation in conservation and archaeology, as well as studying live mammalian cells under the IR microprobe for in situ drug response, an important tool in anti-cancer research. This past year, B22 provided nano- and micro-spectroscopy imaging experiments studying detection of volatile acetone by MOFs (10.1002/admi.202201401) as molecular sensors, understanding host-parasite interactions at cellular and subcellular levels using infrared microspectroscopy imaging (10.3390/cells11050811) , following the photo-oxidation catalysis of methane to methanol (a key reaction for increasing energy density) (10.3390/cells11050811) and application of terahertz spectroscopy for studying zeolitic

Attendees to the S4SAS conference in September 2022.

imidazole framework (ZIF-71), a large, 816-atom porous nanomaterial ideal for gas capture, catalyst and molecular sensing (10.1021/acs.jpclett.2c00081).

B22 acquired two new team members, Dr. Hendrik Vondracek and Dr. Vishnu Muruganandan. Dr. Vondracek will be the Senior Support Scientist overseeing the new atomic force microscopy coupled to IR end-station. Dr. Vondracek was a post-doctoral fellow at the IR beamline (SISSI) at Elletra (Triesta, Italy) with experience in s-SNOM as well as THz research in biological/biofunctionalised system (new and enhanced technical capabilites at B22, respectively). Collaborative calls for IR nanospectroscopy in photothermal tapping and contact modes, plus scattering-SNOM. AFM-IR will be available during the ongoing commissioning of the instrument aiming to a dedicated call for commissioning/collaborative users in early 2024. In addition, B22 published a technical development paper on the application of deformable mirrors to improving the synchrotron source for illumination in IR hyperspectral imaging by Focal Plane Array detector (10.1364/OL.456049). This work demonstrated the potential of beamshaping using a double set of deformable mirrors that will be further developed in collaboration with the machine learning group at STFC (SciM, headed by Dr. Jeyan Thiyagalingam) and Dr. Muruganandan, our new post-doctoral research fellow. Dr. Muruganandan has experience in telescope-based image-processing algorithms for star tracker and multispectral camera, including adaptive optics and algorithms to image and characterise artificial satellites

B23 Update

B23 is our beamline for synchrotron radiation Circular Dichroism (CD) and Mueller Matrix Polarimetry (MMP). B23 uses circularly polarised light to characterise the structure-architecture of complex chiral materials in solution and in solid-state thin films. Chiral materials have a handedness like our right and left hands that are not superimposable, and absorb differently the circularly polarised light generating CD fingerprint ID spectra. The beamline operates two end-stations: module A and B to accommodate a variety of sample environments. Module A operates in the 170-500nm region (125- 500nm for gas phase) utilising an automated 6-cell turret for protein UV denaturation and/or thermal stability assays, a motorised XY stage to accommodate either microfluidic chips for the separation of proteins by diffusion or a custom made 96-cell multiplate to characterise the biomolecules conformational behaviour and ligand binding screening. Since 2020, module B operating in the 190-650nm spectral region is equipped with the Mueller Matrix Polarimeter (MMP) to study the optical (linear dichroism (LD), circular birefringence (CB)) and chiroptical properties (circular dichroism (CD), and circular birefringence (CB)) of thin films of chiral materials such as polymer, biopolymers, optoelectronics, hydrogels, and twisted liquid crystals. For optoelectronic materials, the measurement of CD at 50micron of spatial resolution can inform about the homogeneity of the supramolecular structure, which is strictly related to their efficacy. For biological samples, CD is also used to monitor in microfluidic chips structural changes, drug binding, protein instabilities as a function of temperature, pressure, ionic strength, surfactant, pH, ligand interactions, and ageing.

Pioneered by the B23 team, CD Imaging (CDi) technology exploits a highly collimated, synchrotron microbeam for scanning thin-films of solid materials for MMP measurements. MMP at B23 is the only synchrotron-based instrument with the required sensitivity to guide the researchers on how to improve the properties of chiral materials. B23, through the PBS Prof. Giuliano Siligardi and Dr. Tamás Jávorfi (senior beamline scientist), participate in an international collaboration, The Chiral Materials Team, which was awarded the 2022 Horizon Prize (Stephanie L Kwolek Award) by the Royal Society of Chemistry. The award recognises their discovery that chiral organic materials can control photon and electron spin to high degree, an important attribute for future computer memory, 3D displays and other spintronic devices. This discovery was made possible through the world-leading MMP instrument uniquely operating at B23.

B23 will see the installation of a new monochromator in 2023 that was designed in-house with our engineering support groups and Dr. Tamás Jávorfi and Dr. Giuliano Siligardi (PBS). The upgrade will improve beam stability and spatial resolution for imaging experiments.

I22 Update

The Small Angle Scattering and Diffraction beamline (I22) offers combined Small and Wide Angle X-ray Scattering (SAXS and WAXS) studies on a range of low order biological, natural and synthetic samples. I22 excels at providing structural information on partially ordered materials ranging from colloidal nanoparticles and thin-films to large hierarchical structures such as bone. The I22 Principal Beamline Scientist Dr Nick Terrill, in collaboration with Prof Michael Rappolt from the School of Food Science and Nutrition at the University of Leeds, manage an Engineering and Physical Sciences Research Council (EPSRC) grant to support an offline SAXS facility at Diamond. The Multi-User Facility for SAXS/WAXS (DL-SAXS) provides a Xenocs Xeuss 3.0 instrument operating with an in-vacuum Eiger-2R 1M detector. The facility accepts Peer-reviewed Panel (PRP) proposals for 25% of its available time with the remaining time dedicated to University of Leeds and sample environment development (SED). The SED laboratory is supported by Dr. Paul Wady and is key to enabling new science within the SCM group. SED has already supported experiments studying iron oxide formation on electrode surfaces (Cambridge University), using an acoustic cell to investigate crystal nuclei (Univeristy of Leeds) and cubosome formation and application to targeting cancer cells (University of Leeds).

I22 began an automation project in 2022 that will use robotics to measure samples stored in a sample hotel co-located at the beamline. The beamline end-station required significant renovation to accommodate cabling, sample hotel and tracks that mobilise the robotic arm. This automation project will store ~5.000 samples in capillaries or DSC pans and take advantage of camera lengths during unsociable hours. Users often require different camera lengths depending on the hierarchical scale under investigation. In some cases, on-site users may have a camera length configuration required by a queued sample and the automation will be designed to efficiently take advantage of on-site user specific camera configurations.

Recyclable elastomers that rival environmentally persistent rubbers

Earth Sciences & Environment – Chemistry – Materials Science – Organic Chemistry – Polymer Science

Elastomers are indispensable to the automotive, healthcare and electronics industries. Most elastomers are petroleum-derived and lack viable recyclability or end-of-life degradation options thus contributing to the plastic waste problem. Current research to address the waste problem has developed more bio-based and potentially degradable alternatives, but these still fall far short of matching the performances of commercialised rubbers. A long-standing bottleneck in elastomer development has been designing for high tensile strength without compromising elasticity/stretchability and elastic recovery.

Elastomers based on polycarbonate and polyester chemistries are attractive as 1) many monomers are bio-based, and 2) the polymer-monomer equilibria can be manipulated to recycle the products back to monomers or to ensure total chain degradation

Pursuing high performance with sustainability, researchers at the University of Oxford investigated block co-polymers based on poly(trimethylene carbonate) (PTMC) and zinc-ionomer polyesters. The soft, flexible, yet highly entangled chains of the polycarbonate (PTMC) appealed for stretchability; the harder zinc-ionomer polyesters would provide maximum tensile strength and excellent elastic recovery.

Many elastomer properties emerge from the formation of physically cross-linked networks. ABA-type block co-polymers achieve the cross-linked networks through block microphase separation where *A* = hard rigid and *B* = soft flexible blocks. Small Angle X-ray Scattering (SAXS) measurements using the DL-SAXS instrument at Diamond allowed the researchers to probe the presence of this crucial phase separation behaviour with A = Zn-ionomer polyester and B = PTMC. Moreover, the mechanical performance of the polymer films can now be directly correlated to the precise morphology of the phase separation described by the SAXS patterns.

The results from the University of Oxford and DL-SAXS, showed that the bio-based polyester/carbonate thermoplastics elastomers retained phase morphology on chemical functionalisation and endowed high-performance elastomer characteristics with spherical or cylindrical A-domains dispersed in a rubbery B-matrix. The elastomers match, or out-perform, commercial polyurethane and non-recyclable prevalent rubbers by combining high tensile strengths (60 MPa) and extensibilities (>800 %) with excellent elastic recovery (>95 %). Importantly, the elastomers do so whilst being re-processable, efficiently chemically recyclable back to monomer and/or fully degradable at end-of-life.

The findings can be widely applied to polymer design strategies for adapting or enhancing material properties for innovative application areas or replacing commodity plastics. Notable is the small, yet efficient, quantities of polyester (< 20 wt%) and zinc (< 1 wt%) required with the PTMC polycarbonate to significantly enhance performance. Furthermore, the synthetic approach to making the materials is also highly generalisable to many available and bio-based monomers as it couples in one-pot controlled cyclic monomer ring-opening polymerisation and alternating epoxide/anhydride ring-opening copolymerisation strategies.

Related publication:
Gregory, GL. *et al.* Block poly (carbonate- ester) ionomers as high-performance and recyclable thermoplastic elastomers. *Angewandte Chemie* **134,47**: e202210748 (2022). DOI: 10.1002/anie.202210748

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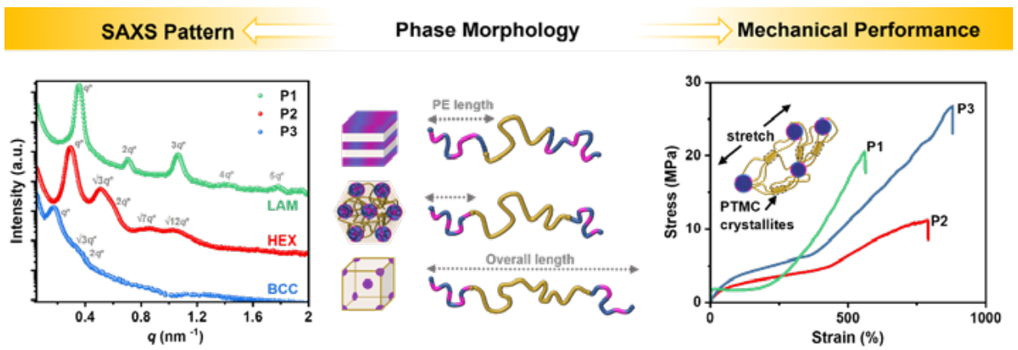

SAXS profiles measured of degradable polycarbonate-block-polyester films were used to assign block microphase separation behaviour for polymers differing in polyester (PE) content or overall molecular weight (P1-P3). This was then correlated to the stress—strain mechanical response. The impressive stress at break values of the materials is attributed to crystallites of the PTMC polycarbonate formed during stretching and reinforcement by interactions in the polyester segments.

Targeting paediatric tumour cells with functionalised cubic phase nanoparticles

Drug Delivery – Non Communicable Diseases – Health & Wellbeing – Cancer – Life Sciences & Biotech

Focused delivery of chemotherapy drugs at tumour sites can increase the effectiveness of the treatments while reducing potential side effects. This long-standing challenge is being addressed using next-generation nanostructured liquid crystalline nanoparticles (NPs) called cubosomes. Cubosomes have high loading capacity, biocompatibility and thermostability, and have been shown to effectively deliver therapeutics to tumour sites in the patient's body. Cubosomes can be designed for active targeting, to direct the therapeutics to the tumour cells, and protect healthy cells from systemic side-effects. Active targeting can be achieved by attaching antibodies, peptides or aptamers, that recognise cancer cells. Furthermore, cubosomes can be designed to respond to external stimuli such as magnetic fields, temperature or pH offering further control over drug delivery.

Researchers from the University of Oxford evaluated an active tumour-targeting cubosome system directed towards rhabdomyosarcoma (RMS) cells. They employed a top-down synthesis approach to produce blank cubic phase NPs, which were subsequently functionalised with hyaluronic acid (HA), anti-CD221 half-sized antibodies (ha-Abs) and superparamagnetic iron oxide nanoparticles (SPIONs). These triple-functionalised cubosomes can be controlled via an external magnetic field.

Small-Angle X-ray Scattering (SAXS) measurements on Diamond's B21 beamline investigated the lipid lattice patterns and verified the formation of cubic phases. Both SAXS and cryo-Electron Microscopy (cryo-EM) performed at the Electron Bio-Imaging Centre (eBIC) played an important role in optimising the cubosome synthesis method and validating the structures throughout the investigation.

The researchers found that all the cubic phase NPs possessed the primitive (Im3m) cubic phase, lattice parameters of 126-155 Å, and water channel diameters of 22-30 Å. The well-organised lattice patterns, however, were compromised by incorporating more than 3% SPIONs. The stability analysis showed that antibody-conjugated cubosomes were able to maintain the Im3m structure for 40 days under ambient conditions, while the integrity of triple-functionalised cubosomes was preserved for 30 days under the same conditions.

The cubosome-based drug delivery platform constructed in this study can encapsulate large quantities of hydrophilic, lipophilic, or amphiphilic therapeutics and confine the chemotherapy to tumour tissues in an active manner. In addition, the cubosome synthesis and functionalisation procedures the team has established may also be useful in structural biology, vaccine, transfection, cosmetics, or biomedical imaging.

Related publication:
Mun, H. *et al.* CD44 and CD221 directed magnetic cubosomes for the targeted delivery of helenalin to rhabdomyosarcoma cells. *Nano Research* **16**, 2915–2926 (2023). DOI:10.1007/s12274-022-5037-4

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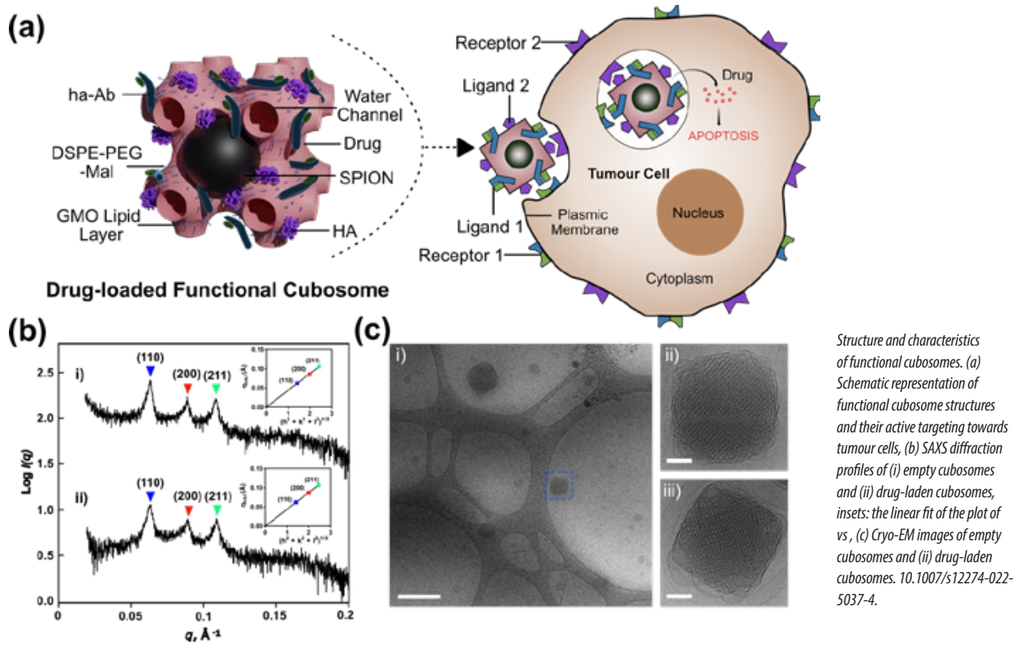

Structure and characteristics of functional cubosomes. (a) Schematic representation of functional cubosome structures and their active targeting towards tumour cells, (b) SAXS diffraction profiles of (i) empty cubosomes and (ii) drug-laden cubosomes, insets: the linear fit of the plot of vs , (c) Cryo-EM images of empty cubosomes and (ii) drug-laden cubosomes. 10.1007/s12274-022-5037-4.

Capturing Sulphur-dioxide in Zirconium-based Metal-Organic Frameworks

Desertification & Pollution – Earth Sciences & Environment – Chemistry – Materials Science – Chemical Engineering – Engineering & Technology – Metal-Organic Frameworks – Metallurgy – Organometallic Chemistry

The growing concern over air pollution has driven the search for new and efficient methods to remove industrial emissions, and desirably to enable recovery from exhaust gases with conversion into chemical feedstocks. Sulphur-dioxide (SO_2) is a major air pollutant with a significant impact on human health. Its highly corrosive and reactive nature generally leads to severe structural degradation in capture materials.

Metal-organic frameworks (MOFs) are porous, crystalline-like materials, and Zirconium-based metal-organic frameworks (Zr-MOFs) have emerged as promising materials for exhaust gases' capture due to their high surface area and tuneable pore environment. Therefore, understanding the interactions between e.g. SO_2 and the pore environment of Zr-MOFs is essential to designing efficient and stable materials for Sulphur-dioxide capture.

By studying the pore environment and its impact on SO_2 adsorption, researchers can develop new principles for the design of MOFs with high Sulphur- dioxide adsorption at both low and high concentrations, enabling their use in a broader range of applications and contributing to the mitigation of air pollution. In addition, the development of regenerable methods for SO_2 capture and recycling of the sorbent material can also reduce waste production and support the sustainability of the technology.

The porous nature of MOFs allows them to capture guest molecules, and host-guest interactions are of fundamental importance. Researchers from the University of Manchester in collaboration with Diamond beamline B22 scientists used a combination of infrared micro-spectroscopy (microFTIR) on Diamond's B22 beamline and *in situ* X-ray diffraction on I11, together with inelastic neutron scattering at the ISIS Neutron & Muon Source to enable the visualisation of the binding domains of adsorbed SO_2molecules and host-guest binding dynamics in Zr-MOFs at the atomic scale and molecular level.

Their results demonstrated that introducing functional groups *(i.e.* -NH_2 and –S-) and atomically-dispersed Cu^{II} sites into a family of Zr-MOFs can effectively enhance the adsorption of Sulphur- dioxide at low pressure. In addition, the confined metal-ligand cages in Zr-bptc offer an optimal pore environment for effective SO_2capture and conversion.

Revealing the role of the pore environment (including pore size, pore geometry and functional groups) and understanding the fundamental host-guest chemistry at the atomic and molecular level making a revolutionary change to the design of the next generation of functional materials.

This work will inform the design of new materials optimised for Sulphur-dioxide capture. These could be applied to storage systems to minimise transport costs and space. In addition, materials with exceptional Sulphur-dioxide capture capability at low pressure will incubate the development of in-vehicle desulphurisation devices.

Related publication:
Li, J. *et al.* Structural and dynamic analysis of Sulphur- dioxide adsorption in a series of Zirconium-based Metal-Organic Frameworks. *Angewandte Chemie International Edition* **61,** 36: e202207259 (2022). DOI:10.1002/anie.202207259

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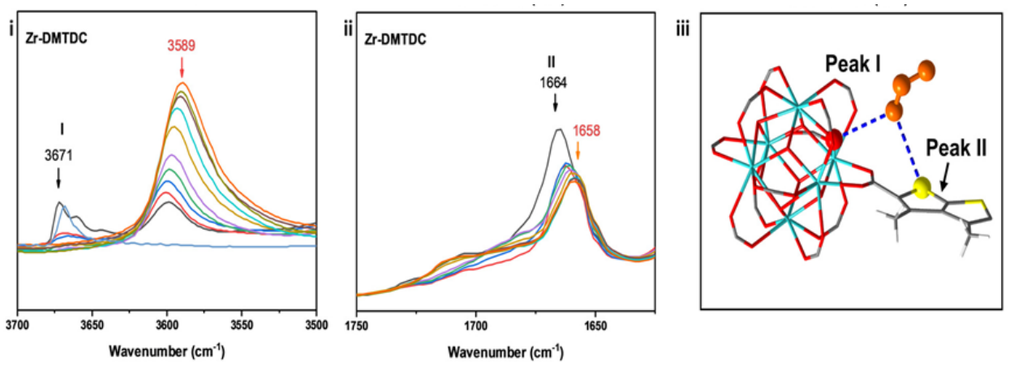

IR spectra of (i) ν(μ3-OH) and (ii) ν(S—C) stretching region for Zr-DMTDC at various loadings of SO_2; (iii). Views of corresponding structures (in various SO_2-loading experiments: black: bare MOF, red: 1% SO_2-loading, blue: 2% SO_2-loading, green: 5% SO_2-loading, violet: 10% SO_2-loading, dark yellow: 20% SO_2-loading, cyan: 40% SO_2-loading, light wine: 60% SO_2-loading, wine: 80% SO_2-loading, orange: 100% SO_2-loading).

Directing lipid nanoparticles for anticancer therapy

Drug Delivery – Non-Communicable Diseases – Health & Wellbeing – Cancer – Materials Science – Nanoscience/Nanotechnology – Life Sciences & Biotech

Nanomedicine is a rapidly growing field that uses nanoparticles to diagnose and treat diseases. Nanoparticles are tiny particles smaller than 100 nanometres, about 1/1000th the width of a human hair. Nanoparticle formulations can be made from a variety of materials, including proteins, polymers, lipids, metals, and inorganic elements. The advantages of using nanoparticles over traditional drugs and therapies include targeting treatments to specific cells or tissues, which can improve the effectiveness of treatment and reduce the risk of side effects. Nanoparticles can also deliver drugs or other therapies to areas of the body that are difficult to reach with traditional methods.

Lipid nanoparticles (LNPs) have attracted enormous interest as drug delivery vehicles; for example, LNPs were essential to the development of COVID-19 mRNA vaccines. LNPs with an internal cubic symmetry, termed cubosomes, are an emerging class of nanoparticles that offer several advantages, such as high encapsulation of cargo and biocompatibility. To date, however, cubosomes have mainly been used for passive targeting, which often leads to off-target toxicity. Their cytotoxicity and biodistribution *in vivo* are largely underexplored, hindering clinical translation.

Researchers from the University of Leeds attached a synthetic antibody, known as an Affimer, to the surface of engineered cubosomes that were loaded with a model chemotherapeutic drug to actively target colorectal cancer cells. They used a range of biophysical techniques to characterise the cubosomes and studied their therapeutic efficacy extensively in colorectal cancer models both *in vitro* (2D cell culture and 3D spheroid models) and *in vivo* in tumour xenograft bearing mice.

After collecting preliminary Small Angle X-ray Scattering (SAXS) data on the Diamond-Leeds offline SAXS instrument (DL-SAXS), the team used Diamond's I22 beamline to characterise the internal nanostructure adopted by the LNPs upon surface functionalisation and drug encapsulation. Using SAXS was essential, as the internal nanostructure strongly correlates to their *in vivo* performance and greatly impacts the LNPs formation-structure-function relationship. The high flux, tuneable energy and spatial/temporal resolution on I22 were crucial, giving diffraction patterns from the weakly scattering dilute samples and resolving the Bragg reflections expected.

The results showed that surface functionalisation and drug encapsulation didn't alter the internal nanostructure symmetry of the LNPs. The cubosomes exhibited preferential accumulation in cancer cells compared to normal cells both *in vitro* and *in vivo*, whilst showing low non-specific absorption and toxicity in other vital organs. Mice subjected to targeted drug-loaded cubosomes experienced: increased drug accumulation in the tumour tissue compared to other vital organs, a decrease in tumour growth, and increased survival rates compared to control groups, demonstrating the exciting potential for Affimer-tagged cubosomes in therapeutic applications.

Understanding how the nanostructure of LNPs leads to function is key to their successful clinical translation. This work focused on engineering LNPs for colorectal cancer treatment. However, understanding LNP structure-function relationships is essential for the development of novel drug delivery vehicles to target a multitude of diseases, vaccines and gene therapy.

Related publication:
Pramanik, A. *et al.* Affimer tagged cubosomes: Targeting of carcinoembryonic antigen expressing colorectal cancer cells using *in vitro* and *in vivo* models. *ACS Applied Materials & Interfaces* **14**, 11078-11091 (2022). DOI: 10.1021/acsami.1c21655

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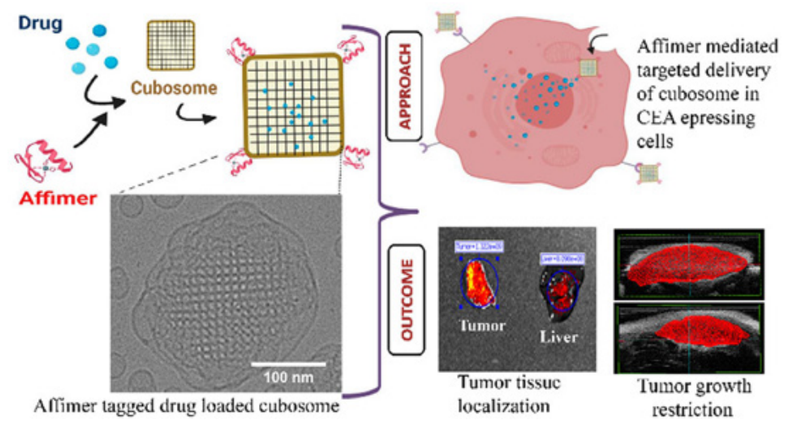

Engineered lipid nanoparticles (cubosomes), loaded with the drug copper acetylacetonate (blue), and with their surfaces functionalized with Affimers that bind carcinoembryonic antigen (red). This potential nanomedicine binds specifically to colorectal cancer cells, restricting tumour growth.

Fine-tuning poly-L-lysine-based antiviral nanomaterials

Biochemistry – Chemistry – Biophysics – Life Science & Biotech – Nanoscience/Nanotechnology

The appearance of new and lethal viruses and their potential threat urgently requires innovative antiviral systems. In addition to the most common and proven pharmacological methods, nanomaterials can represent alternative resources to fight viruses at different stages of infection by selective action or in a broad spectrum. A fundamental requirement is non-toxicity. However, biocompatible nanomaterials often have little or no antiviral activity, preventing their practical use. Carbon-based nanomaterials have displayed encouraging results and can present the required mix of biocompatibility and antiviral properties. Researchers at the University of Sassari recently synthesised a polymeric nanomaterial, derived from the amino acid L-lysine, with an antiviral activity against SARS-CoV-2 and a good safety profile *in vitro*.

The low cost of production and ease of synthesis strongly support the further development of such innovative nanomaterials as a tool for potential COVID-19 treatments and as broad-spectrum antivirals. As the polymer structure is highly dependent on the starting pH conditions and hydrothermal temperature, there is a need to study the polymerisation process of L-lysine as a function of pH growing conditions.

The research group is developing a new generation of lysine-based nanostructures by modifying the lysine branched structure with other amino acids, such as arginine and glycine, whose structure is not yet understood. They used Synchrotron Radiation Circular Dichroism (SRCD) on Diamond's B23 beamline to understand the supramolecular structure of this peculiar class of biomaterials.

They combined these data with the results of complementary techniques, including UV–Vis, fluorescence measurements, Nuclear Magnetic Resonance, Fourier Transform Infrared Spectroscopy, and Dynamic Light Scattering.

The structural analysis of the poly-L-lysine (PLL) obtained after a hydrothermal treatment (HT) at 200 °C of L-lysine showed significant differences in the homopeptide architecture as a function of pH. The polylysine synthesised at low pH is a hyperbranched cross-linked polymer, whereas a high pH allows the formation of linear structures. It is, therefore, possible to tune the synthesis process to obtain cross-linked or linear lysine polymers by modulating the pH of the starting solution.

L-Lysine-based nanomaterials are expected to significantly impact antiviral materials as this study reveals the temperature and pH conditions under which they can be carefully engineered to modulate their size and surface properties to confer specific purposes. The knowledge acquired in this study has enabled the design of very specific L-lysine-based nanosystems that can inhibit the replication of different types of viruses with potential broad-spectrum responses.

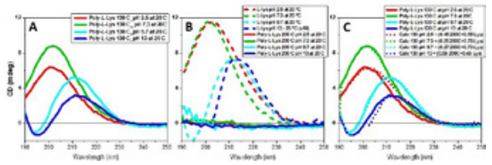

*Figure 2: **A**) CD spectra of HT-130 °C poly-L-lysine prepared from L-lysine aqueous solutions at different pHs (2.5, green line; 7.3, sky blue line; 9.7, red line; blue, 13). The samples have been measured at 20 °C. **B**) CD spectra of HT-200 °C poly-L-lysine prepared from L-lysine aqueous solutions at different pHs (2.5, green line; 7.3, sky blue line; 9.7, red line; blue, 13). The samples have been measured at 20 °C. The CD spectra of pure L-lysine in aqueous solutions at different pH are the dashed lines. **C**) CD spectra of HT-130 °C poly-L-lysine from figure 1a, overlapped to the simulated CD spectra shown as dotted lines. The simulations have been performed by adding different fractions of HT-200 °C PLL (see Fig. 3b) to the corresponding L-lysine aqueous solutions. The CD spectra of L-lysine at pH 13 is normalised because it has been measured with 0.1 cm path length instead of 0.01 cm employed in the other measurements.*

Related publication:

Stagi, L. *et al.* Modulating the polyLlysine structure through the control of the protonation–deprotonation state of Llysine. *Scientific Reports* **12** 19719 (2022). DOI: 10.1038/s41598-022-024109-5

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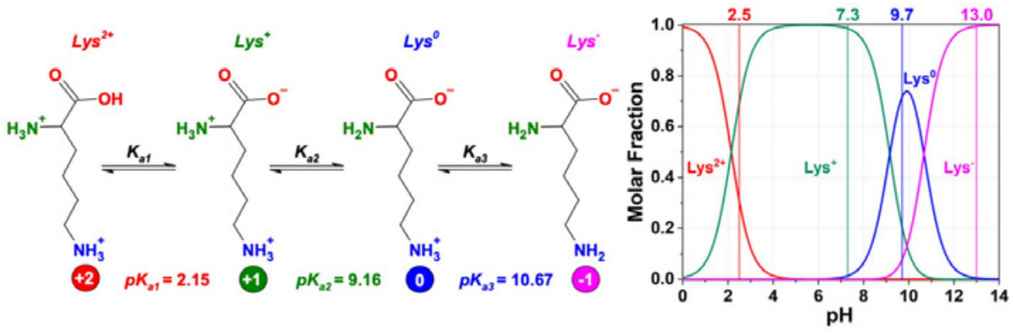


Figure 1: Charges of l-lysine as a function of pH.

Integrated Facilities and Collaborations

As a world-leading centre for synchrotron science and a cornerstone of a world-class site for scientific discovery and innovation at Harwell, Diamond Light Source has powerful synergies with its neighbouring research institutes and beyond the campus, through collaborations and shared visions. The integrated facilities at Diamond present academic and industrial users with a one-stop-shop for research opportunities, enabling them to combine cutting-edge techniques and capabilities to advance their studies. During the period 2022/23, Diamond was active on over 75 grant funded projects (20 of which were Diamond led). Our grant portfolio involves projects with over 65 national and international collaborators, where Diamond has contributed to projects worth over £316m.

The Membrane Protein Lab

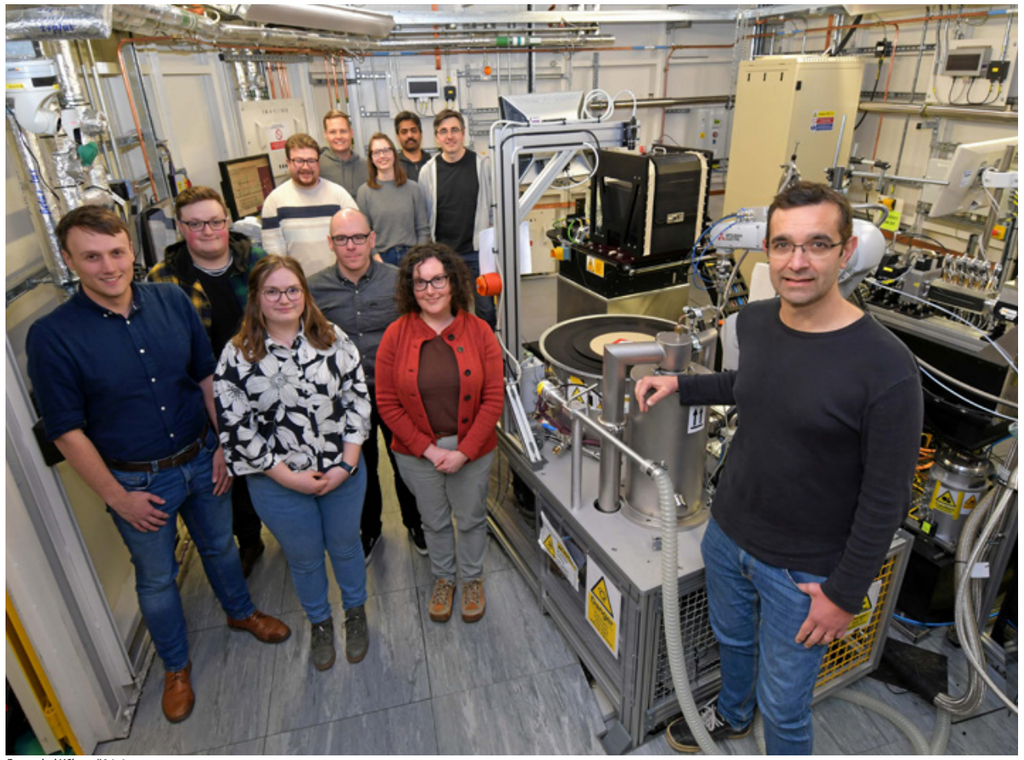
The Membrane Protein Laboratory (MPL), at Diamond is a Wellcome funded resource that supports integrated membrane protein structural biology. Located within the Research Complex at Harwell the MPL enables membrane protein research through the delivery of high-quality samples to Diamond's beamlines and microscopes as well as providing a platform to support membrane protein biochemistry. Membrane proteins are found at the junctions between the outside world and the inner workings of the cell. Multicellular organisms such as humans use membrane proteins for communication, to acquire nutrients and detect threats. Membrane proteins are important targets for biomedicine with over half of all medicines altering membrane protein function. Understanding the structure and function of these proteins in isolation as well as within the wider cellular context will helps us to develop new therapeutics to tackle disease.

Visiting scientists to the MPL can spend anywhere between a day and year in our labs supported by state-of -the art equipment and our experienced support scientists. Working with researchers from Brazil, MPL and eBIC scientists have solved two new single-particle cryo-EM structures from yeast; Ach1p (PDB: 8DH7) and cytochrome C oxidase (complex IV) (PDB: 8DH8) were deposited in the protein databank.

In collaboration with the Rosalind Franklin Institute MPL scientists contributed to a publication in the journal *Science* which looked at new nuclear magnetic resonance (NMR) based methods to quantify interactions between host glycan-proteins and pathogens[1]. Specifically, the authors revealed a distinctive sugar-binding mode mediated by the unusual N-terminal domain from B-origin-lineage SARS-CoV-2 spike protein which is lost in later variants.

In other work MPL and eBIC scientists have published a review of cryo-EM structures of membrane proteins smaller than 100kDa and provided an analysis of the sample preparation routes, data collection methods and processing approaches that were taken[1] in an effort to highlight successful approaches to understanding these difficult proteins.

1. Buchanan CJ, *et al.* Pathogen-sugar interactions revealed by universal saturation transfer analysis. *Science* **377**(6604): eabm3125. (2022) DOI: 10.1126/science.abm3125.



Expanded XChem/I04-1 team.

The XFEL Hub and collaborators in Pohang South Korea in December 2022 at PAL-XFEL for experiments. Left to right: Dr Pierre Aller, Dr Matin Maly, Mr Jack Stubbs, Ms Charlotte Cordery, Dr Ivo Tews, Allen M. Orville, Dr Anastasya Shilova, Dr Sam Horrell, Dr Philip Hinchliffe, Dr Jos Kamps.

2. Harrison PJ, *et al.* A review of the approaches used to solve sub-100 kDa membrane proteins by cryo-electron microscopy. *J Struct Biol.* **215**, 107959. (2023) DOI: 10.1016/j.jsb.2023.107959.

XChem

Installation of the new insertion device on I04-1 has increased working flux by 10x. Alongside the EIGER2 X 9 M detector installed previously, this allows us to collect high-quality data collection in 7.2 seconds exposure time. This has had the most impact on the industrial XChem programme who were previously collecting 60 seconds collections, saving up to 53 seconds per dataset.

The XChem platform has also expanded the sample preparation facility to accommodate increased activity driven by user demand (from both academia and industry), grant funded projects and delivery on new collaborations (EUbOpen and Infratec). Investment in the facility, coupled with growth of the XChem team, enabled a 50% increase in the number of samples produced in Q1 2023 with almost 13,000 crystals mounted for academic user experiments and grant-funded research alone.

Alongside capital investment in the platform, work is ongoing in collaboration with Scientific Software at Diamond to improve the robustness and reliability of the XChem software pipeline ahead of future redevelopment of the software stack to meet the demands of Diamond-II. As part of this work a new deep learning model (Crystal Hits in My Plate (CHiMP)) and user interface (EchoLocator) has been developed to automate crystal identification and selection of coordinates for dispensing compounds.

Following the success of the COVID Moonshot's development of a pre-clinical candidate for the SARS-CoV-2 Main protease, the XChem team and collaborators at Diamond have contributed to two successful National Institute of Health (NIH) grants funding the establishment of Antiviral Drug Discovery (AViDD) Centers for Pathogens of Pandemic Concern: the AI-driven Structure-enabled Antiviral Platform (ASAP) and the Rapidly Emerging Antiviral Drug Development Initiative (READDI). The XChem fragment screening platform is central to both Centers' pipeline for identifying chemical starting points for drug discovery projects. Furthermore, ASAP will also leverage the platform's approach to high-throughput structural biology to enable the rapid development from hit to lead-like molecules. Both centres are also committed to open-science and sharing their data to further enable drug discovery projects and methodology development elsewhere.

Key publications:

Skaist Mehlman T, *et al.* Room-temperature crystallography reveals altered binding of small-molecule fragments to PTP1B *eLife* **12**, e84632 (2023) DOI: 10.7554/eLife.84632

Saar K L, *et al.* Turning high-throughput structural biology into predictive inhibitor design *PNAS* **120**, (11) e2214168120 (2023) DOI: 10.1073/pnas.2214168120

Zaho Y, *et al.* Structural analysis and development of notum fragment screening hits *ACS Chem. Neurosci.* **2022**, 13 (2022) DOI: 10.1021/acschemneuro.2c00325

XFEL-Hub

The XFEL Hub at Diamond continues to provide expertise and support to the UK community engaged in serial crystallography and XFEL-related life science research. This ranges from experimental conception to beamtime proposals, through sample preparations and testing, to XFEL data collection, analysis, and publication. Our Diamond-based activities continue to include organizing and running the block allocation group "Dynamic Structural Biology at Diamond and XFELs" for serial crystallography and time-resolved studies at various MX beamlines at Diamond. This fiscal year, members of the Hub participated in 9 XFEL experiments at the LCLS in the USA, SACLA in Japan, PAL-XFEL in Korea, or the European XFEL in Germany, as well as a site visit to SwissFEL in Switzerland.

Highlighting the synergistic overlap and technology transfer between XFEL and synchrotron facilities, the XFEL Hub spearheads two major projects at Diamond, funded roughly 50:50 by Dr Orville's Wellcome grant and the STFC/UKRI, that will establish methods for time-resolved serial crystallography studies using on-demand sample delivery and reaction initiation strategies that can be correlated with time-resolved X-ray emission spectroscopy (tr-XES) too. Commissioning activities for these two projects are progressing at Diamond beamline VMXi and is a world's first installation at a synchrotron. Our plans also include a collaboration with SwissFEL, which along with the Hub plans to host the Diamond sample delivery capabilities and XES capabilities. The Hub has been testing prototypes for sample delivery and XES data collection with von

Hamos geometry at Diamond. We have analyser crystals to enable XES from copper- and/or iron-dependent metalloenzymes, and an order out for a set of analyser crystals suitable for Ni-containing enzymes.

Dr Orville continues to serve as the life science lead for the UK XFEL project. He delivered a talk at the Royal Society in London on 30 January 2023 as part of the launch event for the Conceptual Design and Options Analysis phase of the project. Comments from the community are always welcome throughout this process.

Active Materials Laboratory

In 2019 Diamond was awarded a grant by the EPSRC to build an Active Materials Laboratory at the synchrotron as part of the National Nuclear User Facilities II scheme to provide more research facilities for nuclear researchers in the UK. The principal aim of this laboratory is to enable Diamond's users to handle active materials on site, either in the short term or performing longer term experiments. All beamline samples will be contained before studying them on one of Diamond's beamlines. Before the laboratory was built nearly

Sofia Diaz-Moreno, Fred Mosselmans, Robin Ibbotson, Chief Technology Officer of Sellafield Ltd, and Adrian Mancuso, Diamond Physical Sciences director.

all radioactive samples had to be brought to site in the containment in which they were going to be measured on the beamlines. This, in particular, made doing experiments in which the sample was heated or exposed to gases on a beamline very hard as the whole cell had to be transported intact. The areas of research that will benefit from the laboratory include materials for new nuclear fission, long-term storage of nuclear materials, a geological disposal facility for medium and high activity waste, how radionuclides behave in the environment and materials for nuclear fusion.

The building was constructed over the period October 2020 – July 2021, before a fit out that was extended due to various Covid-related supply issues. The lab is now complete. It consists of two working labs, one nominally a wet laboratory and the other a dry one. They house two glove boxes, a high temperature furnace, an anaerobic Coy chamber, microscopes, centrifuges and other standard lab equipment. Furthermore, there is a counting room with a Gamma spectrometer and liquid scintillation counter. The Laboratory building also houses a storage room for storing active materials in a safe and secure manner.

In 2022 the lab welcomed its first users, the group of Susannah Speller from Oxford. They are studying the effects of irradiation on high temperature superconductors, that may be used in fusion reactors. Several other groups have used the lab since then and Robin Ibbotson, Chief Technology Officer of Sellafield Ltd formally opened the laboratory in November 2022.

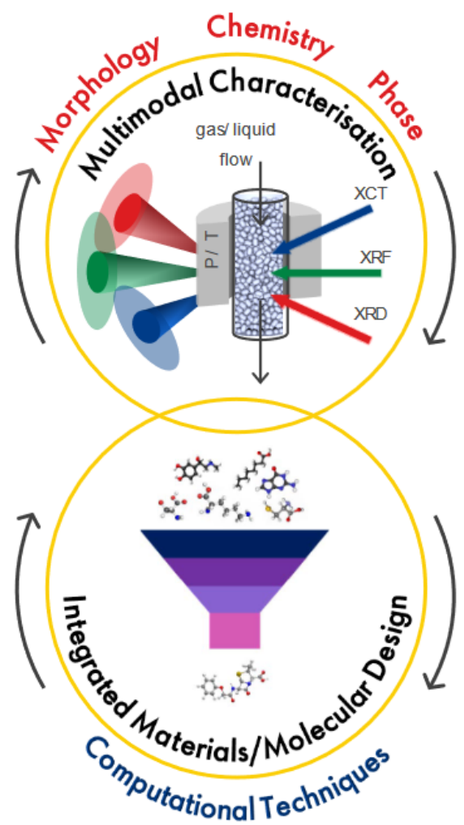
InFUSE: INterface with the Future – Underpinning Science to support the Energy transition

The InFUSE collaboration is a large-scale Prosperity Partnership funded by the EPSRC and SHELL. There are three partners; Imperial College London, SHELL and Diamond Light Source with multiple departments involved across many sites. The aim is to understand and overcome some of the fundamental problems that are hampering the efforts to achieve Net-Zero. Addressing Climate Change on the timescales required needs a rapid translation of technology through close partnership between academia and industry, with a shared vision and commitment.

In this project, the team have identified how important the processes occurring at solid-fluid interfaces are in determining the properties of the system. The influence reveals itself in many relevant science areas such as carbon capture, utilisation and storage (CCUS), electrochemistry (for example at interfaces in battery materials), catalysis and lubricants amongst many others. It is clear that the interface morphology, phase and chemistry all play a role as to how the interface influences the properties.

A complete picture can only be formulated by studying the problem on many fronts, adapting a correlative multi-technique approach, as shown in the Figure. Diamond Light Source contributes to this effort in a number of ways; three of the science groups from the physical science division are actively involved – structures and surfaces, spectroscopy and imaging. Three post-doctoral researchers based at Diamond complement those based in the research groups at Imperial College London. They aim to develop new sample environments that can move between beamlines to simplify such correlative studies, especially in ensuring that *operando* environments expose the sample to the same conditions on the different instruments. Such combined experiments also open up studies at different length scales or processes occurring at differing timescales. Importantly, the InFUSE team is also concentrating on developing modelling and analysis tools, such that interpreting the data from the different techniques will identify the important correlative behaviour.

The sample environments and analysis methodology developed as part of InFUSE will be made available to all Diamond users; at present this includes small cells for catalysis studies at relevant pressures together with improved electrochemistry cells. The InFUSE team is committed to using the techniques at Diamond to optimise the interfaces to deliver improved performance. If you would like further details of the project please contact any of the relevant science group leaders at Diamond, Chris Nicklin, Paul Quinn or Sofia Diaz-Moreno.



The InFUSE methodology combines multimodal characterisation with computational modelling for materials design.

Machine Operation and Development

Richard Walker, Technical Director

2022/23 was our 16th year of operation, and unlike the previous two years which were affected by the pandemic, was carried out entirely in normal operating mode: 6-day running per week, from 09:00 Wednesday to 09:00 Tuesday interspersed with Machine Development days.

A total of 214 days (5145 hours) were scheduled for User Mode operation, including 5 beamline start-up days. All operation was in standard multibunch mode (900 bunch train) with total current of 300 mA, apart from 6 days of "hybrid" mode in May 2022, consisting of a 686 bunch train with a high charge (3 nC) bunch in the middle of the dark gap.

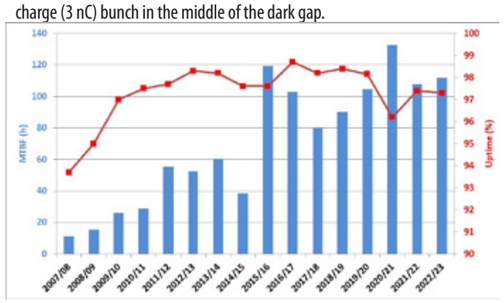


Figure 1. Mean Time Between Failures (MTBF) and Uptime by operating year.

The annual operating statistics are shown in Fig. 1. The overall MTBF continued to be good at almost 112 hrs, and each of the five operating Runs achieved a MTBF in excess of 72 hrs, which is the target minimum. However, the 97.3% uptime was disappointingly below the target of 98%. Around two-thirds of beam trips were recovered in under 2 hrs, but with several longer outages for a variety of different reasons, the longest of which was 9 hours caused by a power outage, the overall Mean Time To Repair (MTTR) was 2.5 hours.

Insertion Device Developments

In June 2022 a specially extended 4-week machine shutdown took place to allow a major modification of straight 4 of the storage ring. Two new insertion devices were installed (see Fig. 2), a 2m in-vacuum cryogenic permanent magnet undulator (CPMU4) for the I04 beamline, replacing the original in-vacuum permanent magnet device, and a 1.6m in-vacuum hybrid permanent magnet undulator (HPMU) for the I04-1 beamline, replacing a 0.7m ex-vacuum permanent magnet undulator. To create the extra space needed for the HPMU, the downstream girder had to be replaced with a new, shorter, girder. Beams from the two undulators were previously separated by a 3-magnet chicane. In order to preserve the same canting angles, with different spacing of the undulators, it was necessary to convert this into a 4-magnet chicane. The modification was a great success with both beamlines reporting significantly enhanced flux compared to previously.

Another major piece of work was the successful upgrade of the cryo-coolers on the first superconducting wiggler which is used for the I15 beamline, which was carried out by Insertion Device Group staff.

Radiofrequency Developments

Further progress has been made with the RF upgrade programme which is aimed at increasing the resilience of machine operation. The third normal conducting HOM-damped cavity has now been commissioned with its 120 kW solid-state amplifier and digital low-level RF system. The amplifier was installed on a platform between Control & Instrumentation Areas as shown in Fig. 3. This is now a complete RF system identical to the ones that will be used for Diamond-II, using similar platforms.

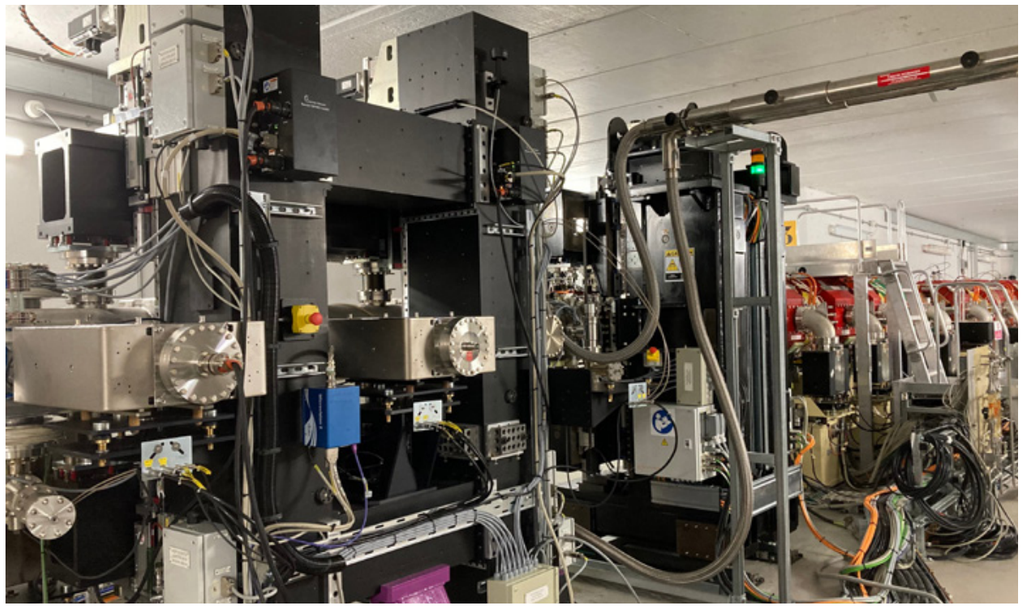


Figure 2. Straight 4 in the storage ring showing the two new insertion devices, CPMU (left) for the I04 beamline and HPMU (right) for the I04-1 beamline. The new girder is visible following the undulators.

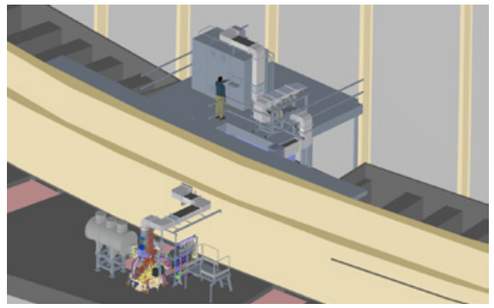


Figure 3. CAD model showing the arrangement of the new RF cavity, its solid-state amplifier and connecting waveguide.

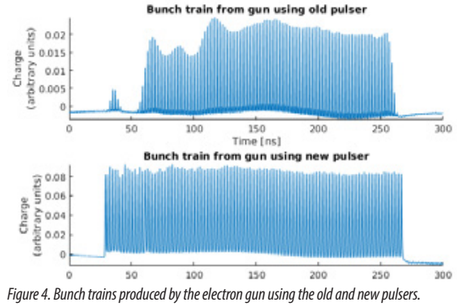


Figure 4. Bunch trains produced by the electron gun using the old and new pulsers.

Another development was the installation of a new pulser for the linac gun which produces a much 'cleaner' pulse train than the old pulser as shown in Fig. 4 and which also allows arbitrary bunch trains to be produced. The next development will be to use the new pulser to carry out multi-bunch top-up in user mode operation of the storage ring, greatly reducing the length of time the beam is disturbed compared to the current single bunch top-up.

Power Supply Developments

The 1200 magnet power supplies used on the Diamond accelerators continue to operate reliably with only one planned intervention that interrupted user operation to correct a fault, and no beam trips during user time. Two new power supplies associated with a CPMU and a HPMU in cell 4 were installed and commissioned.

Development of new digital controller power supply cards continued (see Fig. 5), which will eventually replace the obsolete PSI controller and ADC cards. Twelve were firstly deployed on the Linac to provide operational experience on the actual machine. Subsequently the first production batches of 100 and 160 cards have been delivered and will be progressively installed in different locations, to confirm they can operate with all of the different types of power supply.

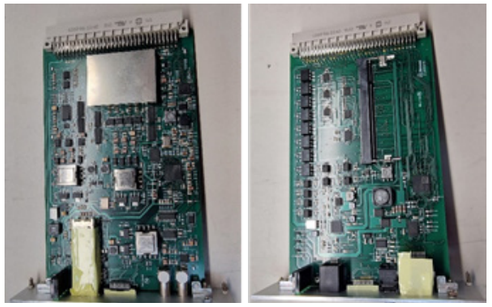


Figure 5. The two new power supply controller cards, the Digital Power Supply card (left) and the System Interface Card (right).

Diagnostics Developments

Single-crystal CVD diamond (sc-diamond) detectors are an established transmissive synchrotron beamline diagnostic instrument used for X-ray beam position and beam intensity monitoring. Silicon carbide (4H-SiC) devices are recently commercialised alternatives which have the potential to provide the same diagnostic information but with a much larger transmissive aperture (see Fig. 6). . The position-sensitive regions for the sc-diamond and 4H-SiC devices are 3mm x 3mm and 9mm x 3mm respectively.

An experimental comparison of the performance of the two devices has been carried out, comparing signal uniformity across the surface of the detectors, kHz intensity measurements, and kHz beam position measurements. Results have shown equivalent operational performance in beam position measurement and flux linearity such that the silicon carbide XBPMs are a promising instrument to add to the suite of diagnostics available to beamlines.

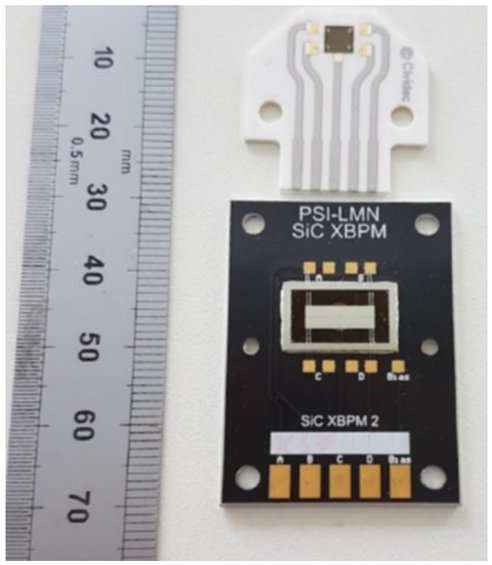


Figure 6: The two X-ray beam position monitors used for this comparison. Top: an XBPM using a 20 µm thick sc-diamond plate; Bottom: an XBPM with a 10 µm thick 4H-SiC frame with 2.3 µm thick window.

X-ray Technologies at Diamond

It is self-evident that for our instruments to produce world-leading science, we need to have world-class optics, detectors and computing technologies at our fingertips; technological advances never stop but are continually evolving. This section describes the support and advances in the Optics and Metrology Group, Detector Group and Scientific Software Controls and Computation department at Diamond Light Source. Advances which are supporting and enhancing our capabilities today are described, but also developments that will keep us competitive over the next few years. These groups are very active in calculations and specifications for beamlines and instruments being put forward and planned for Diamond-II, an integrated upgrade of the synchrotron, beamlines and computational facilities. These advances continue to keep us competitive worldwide, and Diamond is proud to be on the forefront of many of these technologies.

Optics and Metrology Group

Kawal Sawhney, Optics and Metrology Group Leader

Despite the past year's challenges, the Optics & Metrology (O&M) group has kept up a steady pace of research and development. Our activities include improvements of the current beamlines, plans for the Diamond-II upgrade, the development of novel X-ray optics, and the implementation of new metrology techniques. Wavefront measurements using knife-edge scans, a novel technique developed by the O&M group, have already been used for rapid optimisation of the VMXm microfocus mirrors. Fast closed-loop operation of bimorph mirrors has now been made possible by a combination of precise in-house metrology and close collaboration with industry. These are the most prominent examples of the wide-ranging capabilities that O&M continues to foster.

The Diamond-II upgrade remains a major focus of the O&M group's activities. We are refining the beamline designs and determining tolerances on optical components. We are providing Diamond's engineers with power loads on critical components, and we are judging the performance of those components from the engineers' FEA simulations. To make room for the Diamond-II flagship beamline CSXID at I17, a new optical metrology lab OML2 is being built to house the equipment from the current lab, which will be dismantled. The physical metrology laboratory (PML) is being extensively used in collaboration with Diamond's engineers for high-precision tests of prototype motion stages in monochromators and other optical components.

Fast alignment of X-ray focusing optics using wavefront sensing

Mirrors that focus X-rays into sub-micrometre focal spots are used on many beamlines at Diamond. The resulting X-ray beam can be used for studying small samples or for high spatial resolution mapping of inhomogeneous samples. Precise adjustment of the mirror angle and the sample position is essential to get a small focus. A method for rapid alignment would allow the small spot size to be maintained in the presence of drift in beamline optics.

The wavefront is a useful concept for representing the focused X-ray beam. We have developed a new wavefront sensor ("knife-edge wavefront sensor") that is particularly suited to wavefront measurements in focused beam geometries (Fig. 1). An absorbing knife edge is stepped transversely through the focused beam while an area detector positioned downstream measures the X-ray intensity. By independently analysing each pixel and projecting back, the wavefront error at the focusing mirror is determined. A single measurement gives both the mirror angle error and the exact position of the focal plane.

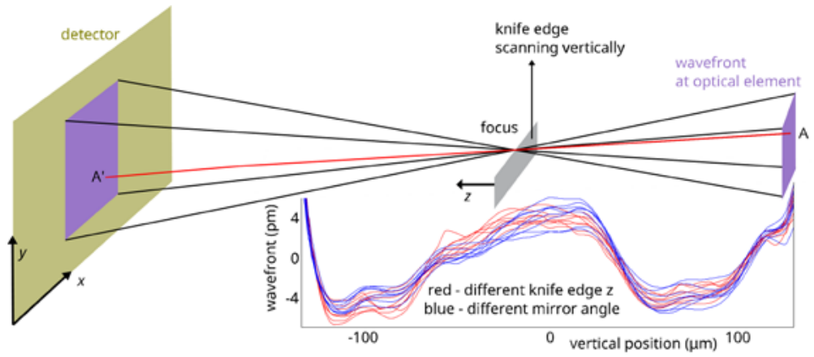


Figure 1: Knife-edge wavefront sensor.

Using the Test Beamline (B16) microfocus mirror system, the wavefront was measured at a range of mirror angles (±30 μrad) and longitudinal (*z*) positions (±3 mm) and the inset graph shows the remaining wavefront error (caused by mirror figure error) after correction for these. Currently, a single wavefront measurement takes about 100 seconds and the data processing 15 seconds using a multi-core workstation. Both these times could however be reduced and then automatic beamline optics realignment at a rate of one per minute could be feasible. The knife-edge wavefront sensor has been employed already on B16 and the VMXm molecular crystallography beamline at Diamond to align the optics and achieve a 400 nm focused beam size.

Fast closed-loop operation of bimorph mirrors

Deformable piezoelectric ("adaptive") bimorph mirrors at synchrotrons have generally been operated at a fixed shape for long periods. Brighter sources now allow larger numbers of samples to be measured in less time. Bimorph mirrors that depend on the piezoelectric effect, which acts instantaneously and generates little heat, can more rapidly match the beam size to the sample. For them to fulfil that potential, however, a concerted effort in many fields has been required: improved designs, strain-free clamping, creep compensation, programmable power supplies, and upgraded metrology capabilities. Closed-loop operation has now been tested at the versatile optics test beamline B16 using feedback from ZPS™ interferometric sensors (Zygo, USA), which can measure mirror figure errors with sub-nanometre resolution at kHz frequencies. In the beamline setup (Fig. 2), the feedback collection does not require any interruption of user operation and permits wavefront errors to be measured simultaneously by X-ray speckle tracking, also developed by O&M. The test mirror was 640 mm long and had 16 electrodes on each side, typical of other mirrors at Diamond. With closed-loop operation at 1 Hz, the focal profile of the test mirror was stabilized within 15 seconds after even a large voltage change, whereas in open loop it took many minutes to stabilize. Beam profiles produced by the test mirror at the focal position were cycled reproducibly at 10 second intervals through a series consisting of the 12 μm FWHM focus, a 55 μm FWHM flattop and a 130 μm flattop. Splitting of the focus into two equal peaks was also demonstrated. This is an important step toward the automated optimisation of adaptive mirrors.

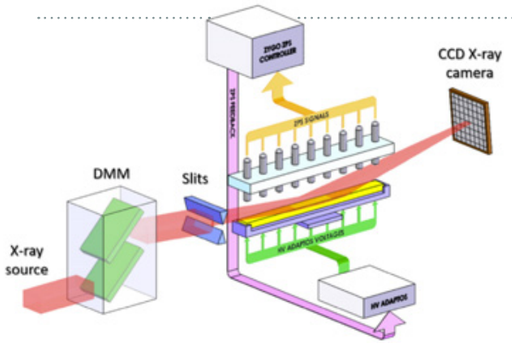


Figure 2: Schematic of beamline setup for testing fast closed-loop operation of deformable piezoelectric bimorph mirror at B16. The double multilayer monochromator (DMM) selected 15.5 keV X-rays.

Mitigation of fine beam structure produced by reflective X-ray optics

X-ray mirrors have always had figure errors that introduce fine structure into the reflected beam off the focal plane. A simple relation of this structure's intensity to parameters of the figure error that manufacturers can measure has long been needed. By combining X-ray speckle tracking wavefront measurements with high-resolution images produced by a modified elliptical mirror and a plane mirror at B16, it has been shown that the intensity at a point in the image is correlated with the curvature error of the wavefront at that point (Fig. 3). A transport of intensity equation showing how the beam profile propagates from the mirror to the imaging detector has been derived. This could allow a given mirror figure error to be tested for acceptably low beam structure in the plane of observation.

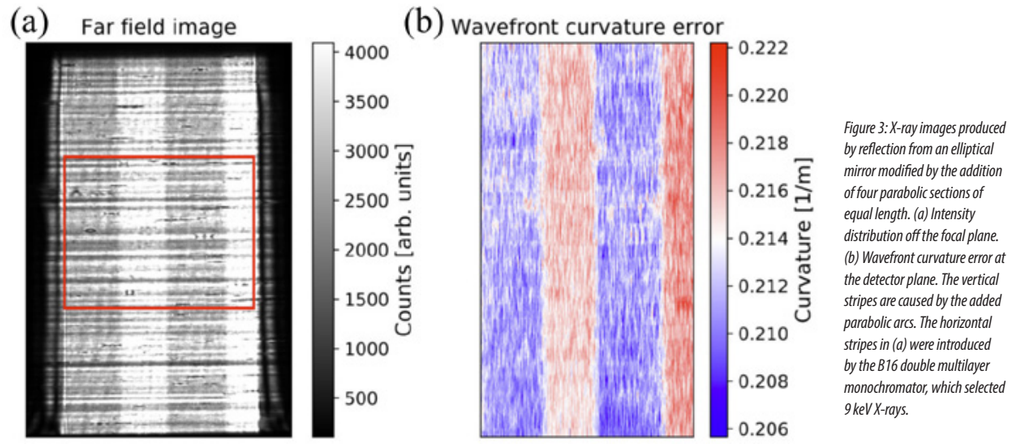


Figure 3: X-ray images produced by reflection from an elliptical mirror modified by the addition of four parabolic sections of equal length. (a) Intensity distribution off the focal plane. (b) Wavefront curvature error at the detector plane. The vertical stripes are caused by the added parabolic arcs. The horizontal stripes in (a) were introduced by the B16 double multilayer monochromator, which selected 9 keV X-rays.

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Quantifying microscale strain in carbon fibre composites using synchrotron X-ray diffraction

Technique Development – Materials Science – Material Engineering & Processes – Engineering & Technology – Composite Materials – Polymer Science

Carbon fibre reinforced polymer (CFRP) design typically relies on material properties obtained from testing small samples or 'coupons' of the material at the macroscale (10 mm+). Although coupon testing is cost-effective and convenient, the tests oversimplify the impact of micro defects, such as voids and wrinkles, that affect the global response of the structures. The current workaround for this issue is to use conservative safety factors and overengineer the composites, which reduces efficiency and performance.

A better understanding of how load is redistributed around defects at the microscale length scales associated with these features will increase confidence in how these artifacts impact material properties and improve input parameters for numerical simulations. However, conventional X-ray diffraction analysis is challenging because carbon fibre is an anisotropic material with a semi-crystalline structure.

As a new methodology was required, researchers at the University of Bath worked with Diamond's B16 beamline to investigate the feasibility of using synchrotron X-ray diffraction for fibre orientation and lattice strain mapping inside CFRPs. The highly flexible setup on B16 and its ability to capture X-ray diffraction and tomography data were crucial to this new approach.

The tests used a sample shaped like a humpback bridge, a standard CFRP geometry with a known solution that induces significant shear stresses between laminates. Its use allows the generation of tensile and compressive stresses and facilitates easy validation with the general loading response of the geometry.

Recording diffraction maps at different loads revealed the residual strains from the manufacturing process and the development of lattice strain in the fibres. The team developed a new two-scale modelling process (mesoscale to microscale) to facilitate numerical verification of the results. In general, the model was a good match to the experimental results, except at the edges where factors that could not be reliably modelled are known to exist.

Their results offer the first quantification of micro-scale lattice strain in CFRPs. This study has provided validation of a powerful new approach to studying the microscale behaviour of CFRPs and other composite materials. Following further validation via comparative methods, which is ongoing, the researchers will be able to use this approach to substantially improve our understanding of the impact of defects and failure modes of CFRPs. That will allow optimisation of the design of these materials, reducing the overengineering of systems and increasing efficiency in numerous applications.

Related publication:
Srisuriyachot, J. *et al.* Carbon fibre lattice strain mapping via microfocus synchrotron X-ray diffraction of a reinforced composite. *Carbon* **200,** 347-360 (2022). DOI: 10.1016/j.carbon.2022.08.041

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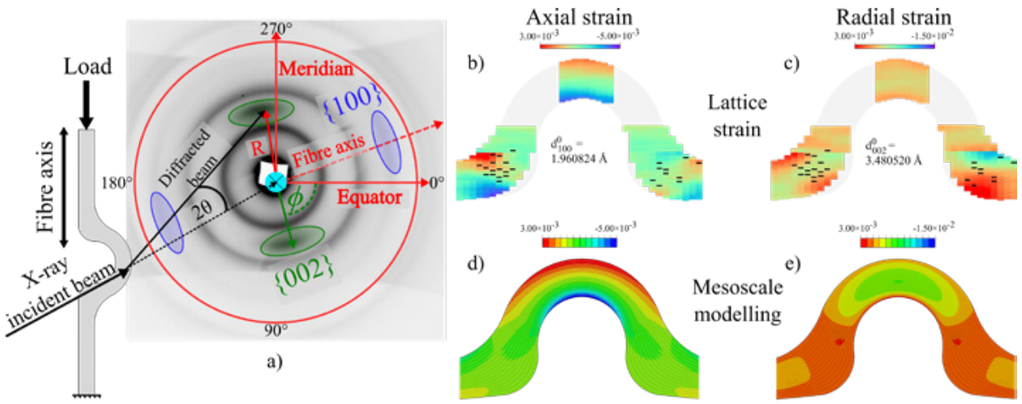


Data collection and key experimental results obtained from in-situ loading of carbon fibre humpback bridge specimen. a) Example X-ray diffraction pattern showing {100} and {002} diffraction peaks which correspond to the axial and radial orientations with the carbon fibre. Lattice strain estimates in the b) axial and c) radial directions determined experimentally from peak shifts associated with these two peaks. Predictions from finite element models which show nominally identical trends in strain in the d) axial and e) radial directions, albeit with localised variations associated with defects in the sample.

Detector Group

Shane Scully, Detector Group Leader

The detector group continues to provide expert operational support to all beamlines and is actively involved in the design and development of multiple new detector technologies. These new detector systems will enhance current capabilities and provide critical improvements that will be needed for Diamond-II. The last year has been one of transition and consolidation for the detector group with several changes in personnel including the previous group leader, Nicola Tartoni, who has been replaced by Shane Scully.

The ARC detector has now been installed in I15-1 with commissioning due to start and continue throughout 2023. This system consists of 24 modules made of electron Schottky CdTe sensors bump-bonded to three Medipix3RX ASICs per module arranged in an arc geometry and was specially developed by the detector group for the X-ray pair distribution function technique enabling faster data collection and a larger Q-range.

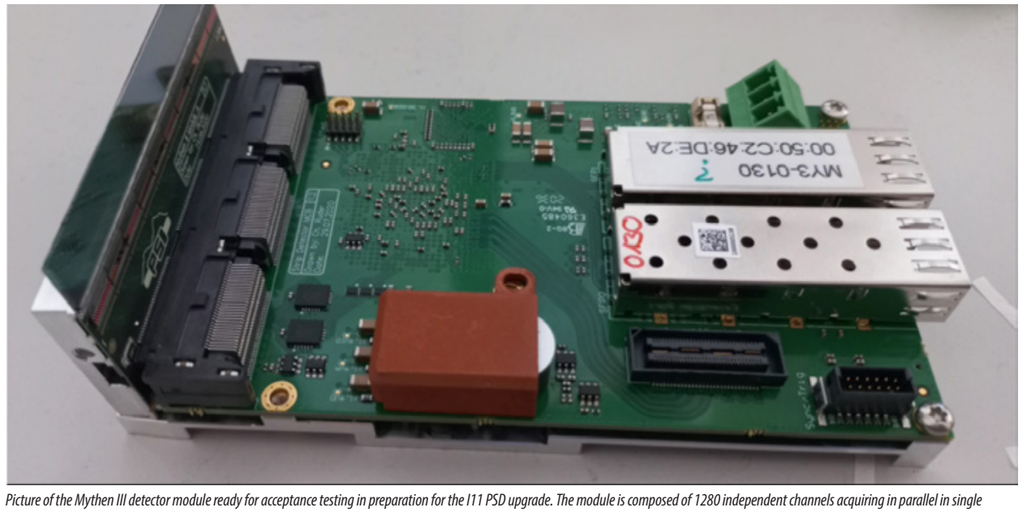
With the Tristan 10M deployed to I19 the group supported I19's time resolved workshop to promote the detector's capabilities to users. The group is now developing a demonstrator for the next generation time resolved detectors using Timepix4 readout ASIC from the Medipix4 collaboration hosted by CERN. This project has developed and started testing a Timepix4 chip board. Timepix4 will build on the foundations established with the Tristan detector development and provide improved capabilities for these time resolved detectors. The XFEL hub has evaluated several detectors, including a Tristan 1M on VMXi for their X-ray emission spectroscopy project. After this successful test XFEL hub have commissioned a modified 2M version and construction has now commenced.

Diamond is performing a leading role in the LEAPS-INNOV work package 2 project to develop two small 10 pixel Germanium sensors for spectroscopic applications. The first sensor from this collaboration recently underwent successful factory acceptance testing and is due to be delivered to Diamond in the next few months. LEAPS – the League of European Accelerator-based Photon Sources – is a strategic consortium initiated by the Directors of the Synchrotron Radiation and Free Electron Laser user facilities in Europe. This is an EU funded collaboration running until 2025. Since 2020, there is also a Diamond-Soleil collaboration in place to develop a 19 pixel small pitch Ge sensor for spectroscopic applications with a different layout, manufacturer and electronic chain.

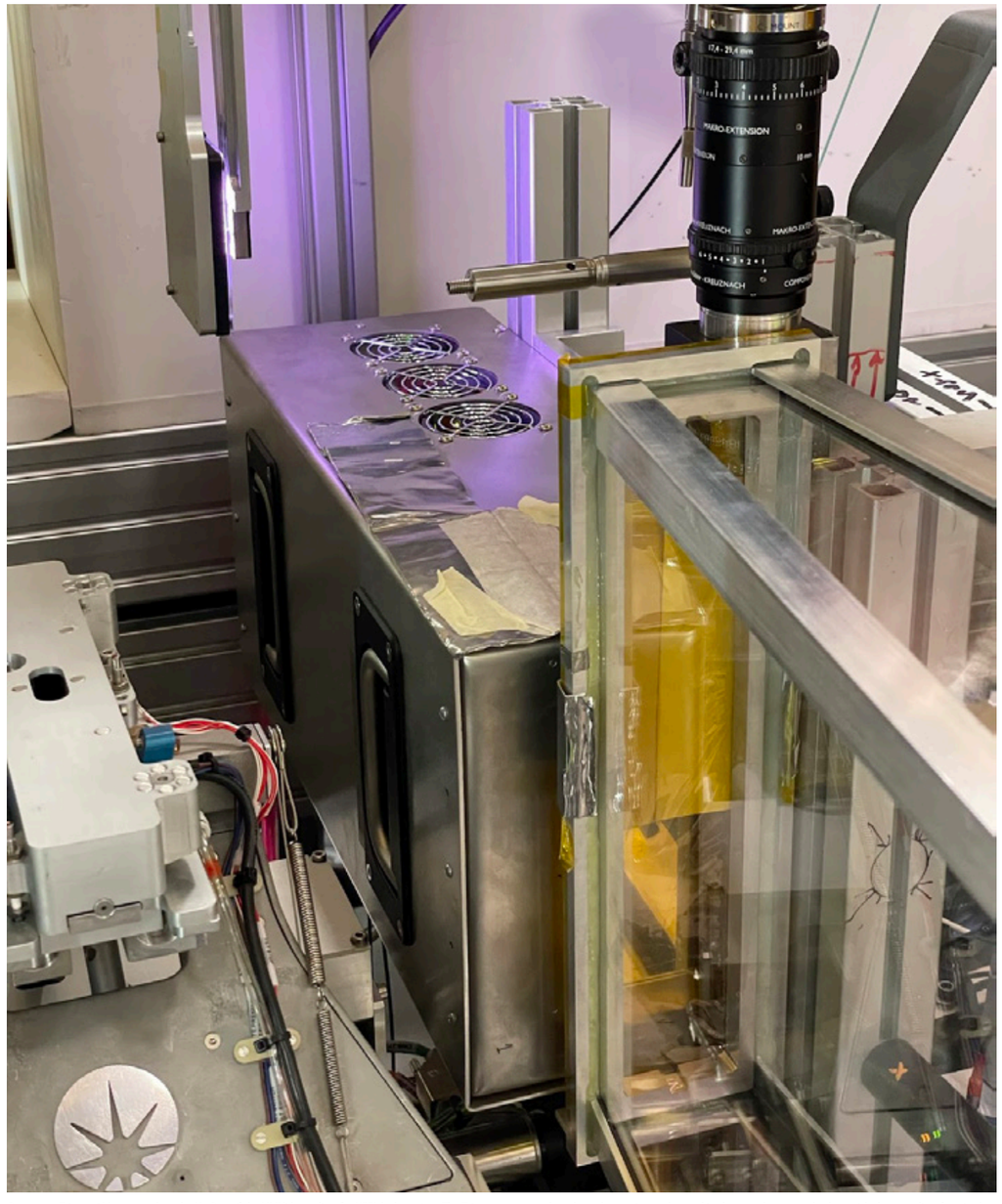
The group is collaborating with STFC in the development of the DynamiX ASIC to meet Diamond-II flux parameters. The aim of this project is to build a high dynamic range charge integrating area detector with hybrid technology incorporating a new charge integrating ASIC and a Cadmium Zinc Telluride (CZT) sensor. This prototype project has started and will run until 2025.

The group has recently taken delivery of a 1M Jungfrau detector system from the Paul Scherrer Institute (PSI) in Switzerland. The Jungfrau is a charge integrating detector capable of measuring high photon rates and incorporates a three stage, gain-switching amplifier in each pixel that can automatically adjust the gain to the amount of charge detected on the pixel. The Jungfrau sticks to the tradition of naming detectors developed at the Swiss Light Source after Swiss mountains. The work to understand the performance of the Jungfrau detector is part of a wider project to develop high performance data handling and analysis for the Diamond-II K04 Ultra-high throughput flagship beamline for MX and XChem.

In the last year work with the I11 beamline and PSI on a project to upgrade the Position Sensitive Detector from the current Mythen-II sensor technology to Mythen-III was also undertaken. The existing PSD has been used by the I11 user community for fast throughput and time-resolved powder diffraction experiments for the order of ten years. Upgrading to the Mythen-III technology provides the opportunity to modify the geometry to a dual row of 28 sensors tiled such that there are no gaps in coverage, improving the scanning time by orders of magnitude by removing the need to move the PSD to cover these gaps. The new sensor module technology will provide lower thresholds through reduced noise, along with a higher rate capability, frame rate and dynamic range. Three independent counters per channel are provided enabling additional modes such as time-over-threshold, energy windowing, fluorescence suppression and pile-up tracking. To date, all 28 modules along with associated electronics have been received and each sensor has received an initial acceptance test using a demonstrator system constructed of a subset of the delivered parts. Characterisation work can now proceed. This demonstrator has also been used to verify the computing and network systems and will be used to develop and test the EPICS infrastructure to streamline the delivery of the full detector to the beamline.



Picture of the Mythen III detector module ready for acceptance testing in preparation for the I11 PSD upgrade. The module is composed of 1280 independent channels acquiring in parallel in single photon counting mode, each connected to a strip of the silicon sensor (shown on the left of the image).



Prototype Tristan 1M mounted on the He Box of the VMXi beamline for XFEL hub testing. The Tristan sensor and readout electronics are housed in the metal rectangular box with three cooling fans in the middle of the image. Tristan is an in-house designed detector for time resolved experiments based on the Timepix3 chip. A modified Tristan 2M is now being produced for XFEL hub.

The Percival Project is another collaboration between Diamond and several institutions (DESY, RAL/STFC, Elettra) for the purpose of designing, developing, and commissioning a prototype of a multi-megapixel soft X-ray CMOS imager with frame rates up to 120 Hz and high dynamic range. The group has received two sensor heads to date with both frontside and backside illuminated test sensors and is designing a vacuum chamber for verifying the sensor performance in beamline measurement conditions.

The Detector Group supported Diamond's contribution to STFC's Stargazing at RAL by providing a live cosmic ray detection demonstration using one of Diamond's Merlin detectors.

Introduction to Scientific Software, Controls and Computation

Scientific Software, Controls and Computation (SSCC) department manages all software, computing and control systems to facilitate and support the science programme of Diamond. The department functions as nine groups: Scientific Computing, Data Analysis, Data Acquisition, Beamline Controls, Accelerator Controls, Electronic Systems, Scientific Information Management Systems, Diamond-II Integrated Software and Cyber Security. The overall structure and function of these areas recognises the importance of, and is optimised to provide, the best possible delivery and support for software, computing, and control systems.

Over the last year there has been an increasing emphasis on planning for Diamond-II. SSCC will deliver new software, control systems and computing as part of the machine upgrade and beamline development for Diamond-II. In addition, it was recognised that there needed to be developments in the underlying software and computing capabilities to prepare for the substantial increase in data rates that will come with Diamond-II. This has been addressed through the design of a new software architecture for photon beamlines and the definition of an extensive core software and computing programme for Diamond-II to deliver new enabling capabilities. (See below: Developing the Diamond-II Core Software and Computing).

Diamond produced more than 10PB (a PB of data is equivalent to 213,000 DVDs) of data in 2022 from photon beamlines and electron microscopes. To support increases in data rates an additional 10PB of first level storage has been procured and along with 24 additional computing nodes (960 cores). It is recognised that provisioning all computing services within Diamond is not sustainable, so work is ongoing to decouple applications and services from the existing computing infrastructure by using containerisation technologies. This will enable the containers to be deployed on both private and public cloud infrastructure. (See below: The Journey to Cloud Native.)

As experiments conducted at Diamond produce increasingly large and complex data sets, it becomes more difficult for users to transport their data back to their home institute and process it. To address this Diamond is increasingly providing users with data processing services to enable information to be extracted from their data. To facilitate this Diamond develops and maintains a suite of data analysis applications to support the photon science and electron microscope programmes. These same tools can also provide near real-time feedback to the user as their experiment progresses. However, some tools are computationally demanding and there is a programme to accelerate the processing using faster computing technologies, such as GPUs, to provide near real-time feedback during experiments. (See below: Accelerating Ptychography Reconstruction Codes.) Advances in toolkits for applying Artificial Intelligence and Machine Learning (AI and ML) techniques have evolved considerably in recent years. They now provide an important opportunity to automate data reduction further and to improve data analysis. (See below: Applications of AI and ML to Diamond Science.)

Historically, user facing applications have run within the graphical user interfaces based on the host operating system. These are now being superseded and made obsolete by web-based technologies. Web based technologies and communication protocols have now evolved sufficiently to now enable the level of performance required for scientific and engineering applications. So, a programme of developments to migrate these applications to web-based environment is ongoing. (See below: A new Engineering Web User Interface.)

Developing the Diamond-II Core Software and Computing

Significant advancement of Diamond's software and computing capability will be required to extract data optimally from the Diamond-II upgrade, and so maximise the scientific opportunity and knowledge gained. Developments will be required to:

- Handle faster detectors and deliver rapid data processing and reduction;
- Support greater automation of experiments, data reduction and analysis;
- Introduce and develop new data processing techniques, including exploitation of AI/ML;
- Provide a more open software environment, facilitating greater collaboration between software and scientist;
- Address obsolescence and modernise the beamline software stack;
- Adapt for changing needs and expectations of Diamond's users.

The Diamond-II core software and computing project will deliver developments focused across key areas identified in Summer 2021 and explored in depth since, covering:

- High Performance Sample Stages;
- Detector Readout, Data Compression and Reduction;
- Modernisation of Data Acquisition Software Framework;
- Science Specific Data Analysis Software Developments;
- Data Archiving;
- Post-visit Data Analysis Services;
- User Administration and Information Management.

The project is one of the five pillars of the Diamond-II programme and is described by a work breakdown structure (WBS) and six work streams: Hardware Infrastructure, Software Infrastructure, Data to Information, Real-time Data, Experiment Management and Information Management. These are hierarchically decomposed into over thirty detailed work packages. A project delivery plan based on phased and early migrations, where appropriate, has been developed. So, whilst the ultimate ambition of the project is to harness the brightness of the new Diamond-II machine and enable new flagship capabilities, this project will realise a continuous stream of incremental benefits to Diamond before this era, reducing technical debt, addressing critical obsolescence, and deploying new capabilities with greater flexibility and extensibility. These developments were documented in the Diamond-II Core Software, Controls & Computing Technical Design Report which was released in March.

The project has enjoyed early success with the roll-out of a new web-based engineering user interface to the Machine. The development of a new Acquisition Platform, is underway with initial core services deployed in a test beamline. A series of projects have explored live data streaming to move from existing "serial" to live "on-the-fly" setups for Ptychography. Live analysis of X-ray diffraction data for experiment feedback is a critical part of the macromolecular crystallography unattended data collection workflow. Initial work has proven the viability of GPUs to reduce image processing time with verified identical algorithmic results.

The Journey to Cloud Native

Diamond is transitioning to a Cloud Native architecture across its scientific computing infrastructure, utilizing key Cloud Native enabling technologies such as containerisation and microservices design patterns. These software encapsulation and development methods drive higher speed and agility in software development, deliver higher application reliability, and provide more portable code. Ultimately, these methods will allow Diamond to develop and deploy software more quickly, decrease time to science, and increase our ability to respond to scientific drivers, opportunities and collaborations.

The most important technology in this landscape is containerisation, which Diamond is adopting heavily in several areas:

- **High Performance Computing (HPC)** - Adoption of a HPC specific containerization technology is enabling off-premise computing for lower priority data processing. By using containers, codebases are no longer coupled to Diamond's on-premise infrastructure and can be easily executed on other public or private off-premise Cloud services (such as the STFC's Openstack Cloud - IRIS).
- **Web Applications** – Web based applications are the most common target for Cloud Native architectures and tooling. Diamond has a number of production, in-house developed and third-party web applications running in containers. These include user interfaces to scientific software, as well as stand-alone data analysis notebooks such as Jupyterhub.
- **Distributed Control Systems** – Both beamline and accelerator controls groups are now running production applications in containers, thereby delivering a consistent bootstrapping environment enabling control and communication with embedded devices.
- **Data Analysis Microservices** – Data analysis middleware, deployed as a microservices application in containers, now underpins all MX data processing pipelines. In turn this is monitored using Cloud Native tools such as Prometheus, Grafana, and Alertmanager to surface and report any issues in the operation of the workflow and so maintain operation of these key services.

Diamond is currently running thousands of containers across the areas listed above. To achieve this in a reliable way, an on-premise Cloud infrastructure has been deployed specifically for container execution and is based on the industry standard Kubernetes container orchestration system.

The Diamond Kubernetes Cloud has approximately 3000 CPU cores deployed as a cluster of machines for high availability and scalability, and hosts a number of production applications that are essential to the operation of Diamond.

Currently Diamond deploys one large Kubernetes cluster which services the whole organization. During the coming year, Diamond will investigate how multiple clusters can be operated, with a view to providing a cluster per beamline. This will present a performance, failure, and administrative domain for each beamline, and is the preferred deployment architecture for Diamond-II.

Accelerating Ptychography Reconstruction Codes

Ptychography is an increasingly important quantitative high-resolution imaging technique used across multiple Diamond imaging beamlines (I08, I13, I14) as well as the electron microscopy facility (ePSIC). Unlike conventional microscopy, ptychography is a lens-less imaging method in which a series of diffraction images (patterns) is collected while raster-scanning an object with an X-ray or electron beam. Using iterative phase retrieval algorithms, this collection of diffraction patterns is then converted computationally into a reconstructed high-resolution image of the object.

The time taken to perform this reconstruction results in a gap between the experiment operation and being able to view an image, and limits our ability to understand the samples under study during the experiment in near real-time. There is also a drive to use this technique to look at larger samples and more dynamic experiments, so it is critical to minimise the time needed for the reconstruction process. Diamond, in collaboration with the Ada Lovelace Centre, has invested in accelerating the ptychography reconstruction codes that are used across the different beamlines and instruments, which includes the *PtyPy* and *PtyREX* software. Most ptychographic iterative phase retrieval algorithms include many steps that can be performed in parallel and are therefore suitable for distributed processing approaches and for harnessing the computational power of modern GPUs.

To demonstrate the impact of code acceleration in ptychography, an example data set has been taken with 1257 diffraction patterns each of size 256x256 pixels collected within 74 seconds at the beamline I08-1 instrument for Soft X-ray ptychography. 200 iterations of the difference map (DM) algorithm as implemented in *PtyPy* resulted in a high-resolution reconstruction of the nanometre-sized structure of a ground scale from a female *Junonia orithya* butterfly wing (Figure 1 below).

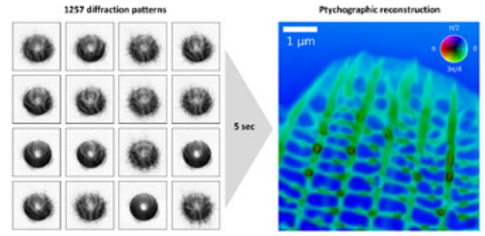


Figure 1: Within 5 seconds, a series of 1257 diffraction patterns are converted by GPU-accelerated ptychography code into a high-resolution image. The colour wheel represents absorption contrast as brightness and phase contrast as hue (color).

Running this reconstruction on a single CPU would take 1736 seconds but distributing the work over multiple processes reduces the time to 114 seconds for a single node on Diamond's Hamilton HPC cluster with 40 CPUs per node. To reduce the time further, we have implemented a GPU-accelerated version of the DM (and other) algorithms using raw CUDA kernels for all major parts of the algorithm wrapped together in Python using *CuPy*. Running on a single HPC cluster node with 4 NVIDIA V100S GPUs reduces the reconstruction time to just 5 seconds – a performance boost by a factor of 22 compared to 40 CPUs.

GPU-accelerated ptychography code has enabled us to implement efficient auto-processing pipelines for ptychographic reconstruction and has substantially improved the experience for beamline users.

Applications of Machine Learning and Artificial Intelligence to Science

Recently there have been significant advances made in the fields of Machine Learning (ML) and Artificial Intelligence (AI), which have attracted widespread interest. Both independently, and in collaboration with others, Diamond has sought to embrace these advances for the facility

In Life Sciences, machine learning tools have been developed to help beamline scientists, and Diamond users, to automatically monitor experiments in which protein crystals are grown. Obtaining diffracting crystals of protein is often a challenging task that requires performing hundreds of experimental trials that are monitored by repeatedly taking microscope images of the small liquid droplets that are used as a growth environment. To automatically monitor these experiments, a set of tools has been created, collectively known as CHiMP (Crystal Hits in My Plate), that utilise deep learning neural networks. The first type of tool classifies the microscope images into different classes such as "clear droplet" or "crystals" giving a quick overview of the progress of the experiment. The second tool pinpoints the location of any crystals and the surrounding droplet in an image, thereby allowing automated targeting of the X-ray beam for collection of diffraction data from the crystals whilst still in the droplet. These tools are currently being utilised on the VMXi beamline enabling fully autonomous operation.

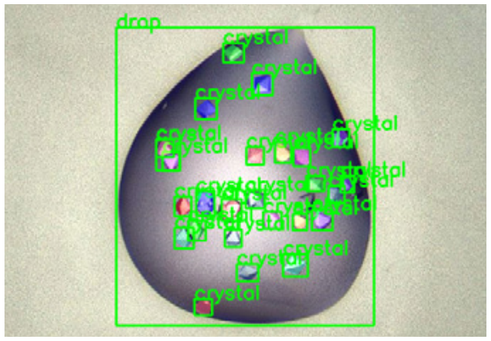


Figure 2: Output of a deep learning network trained to detect the location of drops and crystals in images.

In Physical Sciences the community has sought to focus more upon on tracking and optimising both measurements and experiments with projects such as the Autonomous Formulation Laboratory, (AFL), (DOI: 10.1021/acs.chemmater.2c03118), and gpCAM (DOI: 10.1038/s41598-019-48114-3) and Summit (DOI: 10.1002/cmtd.202000051). These projects aim not only to optimise experimental parameters but also interact with data acquisition systems enabling autonomous experimentation. The AFL project a platform that allows for the optimisation of samples made from stock solutions primarily from small angle scattering data but also other techniques such as spectroscopy and rheological measurements. gpCAM optimises the sample scanning by focussing scan point density on more 'data rich' areas of a target scan area and weighting away from scanning 'data poor' areas. (Figure 3 below). Finally, Summit is a framework for optimising chemical reaction conditions, evaluating and comparing different reaction strategies against each other to present a broader picture to the user. As can be seen, all these packages afford benefits to scientists whether on a beamline, in the lab or simply highlighting a more optimal data collection strategy.

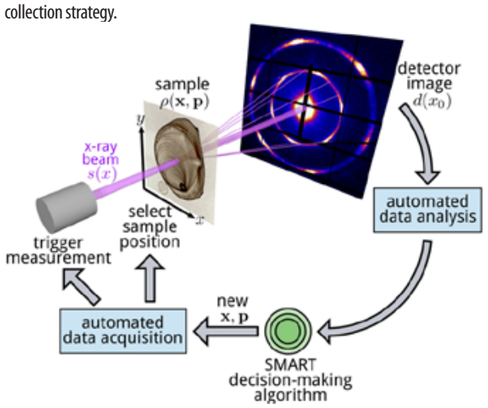


Figure 3: An overview of a data density driven scan from the gpCAM system (Image from Noack et al. – DOI: 10.1038/s41598-019-48114-3, work is under a CC BY 4.0 Licence).

A new Engineering Web User Interface

Software provides the User Interface (UI) for the operation of Diamond; from the control systems, providing the interfaces: to the underlying hardware and equipment, to the data acquisition systems for experiment control, and to the tools for analysis and cataloguing of the collected data. Facility users rely on UIs to make effective use of Diamond.

As part of the Diamond-II software and computing programme, there is a plan to enhance UIs to provide a more integrated experience across the whole suite of Diamond software. This will be achieved by using web technologies. This approach will: make use of modern technologies and benefit from the latest developments in user interfaces, improve the user and developer experience of working with the UIs, and enable the software to be operated remotely, which becomes increasingly important.

The first area where this approach will be applied is the Engineering UI, which is the interface to the control system, used by experts to configure and operate the accelerators and beamlines. This will be needed in the commissioning and operation of Diamond-II and needs to address obsolescence in the existing engineering UI technologies.

The new engineering UI will be based on established web frameworks. Similarly, to the web applications used every day, it is made up of two parts, a client and a server. The front-end, running in a browser, acts as a client and is responsible for the final rendering of the interface, while the server is responsible for delivering correct, timely and up to date data to be rendered. The use of web technologies is ubiquitous in society now, and there is familiarity with its function and behaviour. It is also the maturity and performance of modern web frameworks and toolkits that make this an obvious choice to deliver the next generation of control interfaces.

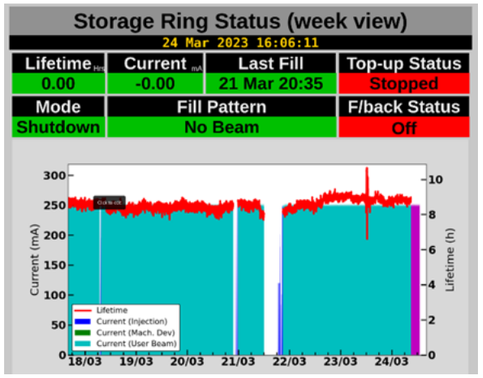


Figure 4: Storage Ring Status Display realised as a Web UI.

The UI front-end is written in TypeScript and makes use of the React framework for rendering and Redux for management of state. The server is written in Python and communication between them is over Websockets.

The scope of this project is to deliver browser-based client software to render the engineering UIs to develop the backend server software that can deliver data to the clients in a performant manner. This means that the server software needs to be able to manage many users connecting and demanding a wide range of data to be displayed. This variability of demand is ideally met by deploying the application in containers orchestrated in Kubernetes (See above: The Journey to Cloud Native).

A working prototype has been developed and have been used for some initial performance testing and analysis. The prototype has been reviewed by external experts to confirm that its implementation is based on solid future-proof technologies that will deliver a high-performance solution that will be fit for purpose for many years. The first roll out of a pilot project delivers Storage Ring Status displays around Diamond (Figure 4 above). Over the coming year further preliminary work will be completed including: additional functionality in the pilot project, documenting a full set of requirements from users, and improving knowledge in key areas. Over the next three to four years, the software will be developed further to deliver a reliable and highly performant interface across the accelerator and beamline control systems. This development will underpin the user interfaces for the higher-level control and data-analysis software. The benefit for Diamond will be one common code base supporting all applications.

Buildings, Infrastructure & Energy Savings

Pete Coll, Head of Installation & Facilities Management Group

Figure 1: Optics Fabrication Building.

Expanding Facility

Since opening in 2007, the Diamond facility has continued to steadily expand. By 2017, I12, I13, I14 (including eBIC/ePSIC) and I21 external buildings had been constructed with the Active Materials Building completed in 2021. Diamond's Installation and Facilities Management (IFM) Group have played a major role in either managing design/construction activities directly or assisting external consultants to deliver these facilities.

A further expansion followed with the completion of the Optics Fabrication Building (OFB) in July 2022 (see Fig. 1). The OFB is a 350m^2 steel framed building with insulated cladding and a mono pitch roof. Foundations were made up of screw piles with cast in-situ concrete ring beams to support a block and beam floor, these work being undertaken during shutdowns to minimise vibrations to the Synchrotron with the rest of the construction work (cladding, screeding, M&E work, flooring and decorations) carried out during operations/shutdowns.

As part of Diamond-II, the Optics Metrology Laboratory (OML) needed to be relocated to allow space to build the CSXID flagship beamline. The new Optics Metrology Laboratory 2 (OML2), located in Zone 4, was recently completed (see Fig. 2). Works will soon commence to relocate equipment from the present OML into the new facility. Demolition of the old laboratory can then begin together with modifications to B16 creating the space required on the Experimental Hall floor for the CSXID beamline.

Figure 2: Optics Metrology Laboratory.

Figure 3: Artist's impression of complete solar panel installation.

Infrastructure Upgrades

As Diamond's facilities grow, there has been a need to expand and upgrade the mechanical and electrical infrastructure to support the expansion. Over the years, there have been significant modifications carried out on the chilled water system including new chillers/adiabatic cooling and introduction of additional cooling water circuits. The high voltage infrastructure has also recently been upgraded to accommodate Diamond's expansion for at least the next ten years.

Due to quality issues in the initial synchrotron roof build resulting in significant deformation, a new roof was needed and has now been installed. Diamond also took the initiative and opportunity of installing solar panels as part of the replacement. The project entailed the removal ~32,000 m^2 of sheeting material, replacing all support fittings, replacing/enhancing the insulation and replacement of the top sheeting before the installation of 2.7 MWp of solar panels (see Fig. 3) which will deliver ~ 2.3 GWh of electricity per annum, being approximately 5% of Diamond's annual electricity consumption.

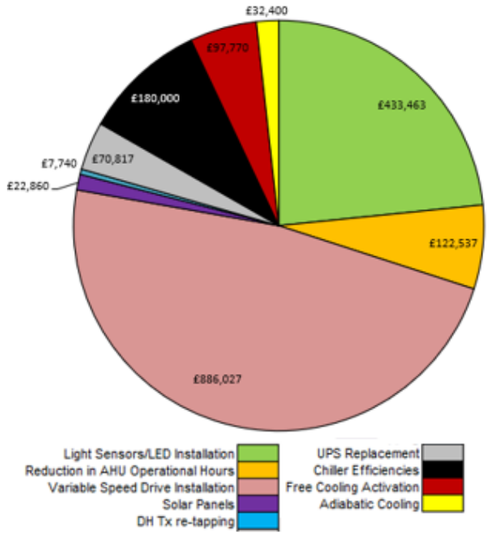


Figure 4: Annual savings by activity for 2022.

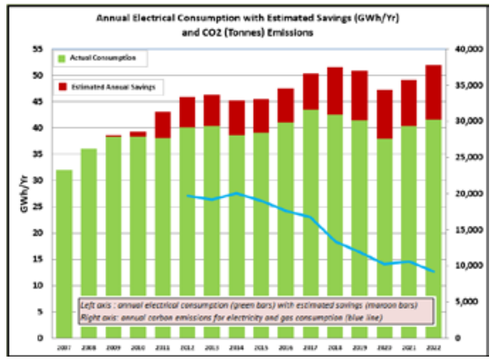


Figure 5: Annual Electricity Consumption with Estimated Savings (kWh/Yr) and Emissions (Tonnes of CO2).

Energy Savings

Since 2010, Diamond has matured its energy saving programme culminating in a total estimation of electrical energy savings of £9.3 million to date, with the highest amount of savings being recorded in 2022 at £1.8 million.

The main contributors to electrical energy savings have been the introduction of light sensors, changing fluorescent lights to LEDs and the installation of variable speed drives on numerous fan and pump systems (thus allow 'turn down' in the circulation of unnecessary air/water). The pie chart (Fig. 4) shows annual savings per category for 2022 (excluding the projected savings from the solar panels on the Synchrotron roof).

Savings compared with actual electrical consumption are shown in the left axis in Fig. 5. The right axis shows the reduction of carbon emissions as a result of these energy saving measures in conjunction with the use of 'greener' electricity, the government's target being to drive for full decarbonisation of the National Grid by 2035.

Diamond's Environmental Sustainability Strategy

Diamond SHE Group, in consultation with Diamond employees, has developed an Environmental Sustainability Strategy and action plan, aligning with Diamond's Ten Year Vision, which has sustainability at its core and is geared towards achieving this across its operations. The strategy is guided by the UN's Sustainability Development Goals and Diamond is fully committed to the Paris Climate agreement – 100% carbon neutrality by 2050. At Diamond we are optimistic and proud of our contribution to supporting research and innovation that is developing solutions to better understand and address global environmental sustainability challenges.

Diamond has a strong track-record in enhancing its own environmental performance. Our Safety, Health & Environment Policy states that the effective management of Environmental matters is of prime importance to the organisation. As such, we undertake to provide an environmentally sound workplace. This includes a commitment to continuous improvement in environmental performance and the setting of objectives.

The recently developed strategy sets out bold ambitions and priorities to further enhance operational environmental performance and better support research and innovation that has a positive environmental sustainability impact.

By pursuing extensive sustainability goals, we are not only acting responsibly toward the environment and society – we are also ensuring the long-term sustainability of Diamond as a national facility.

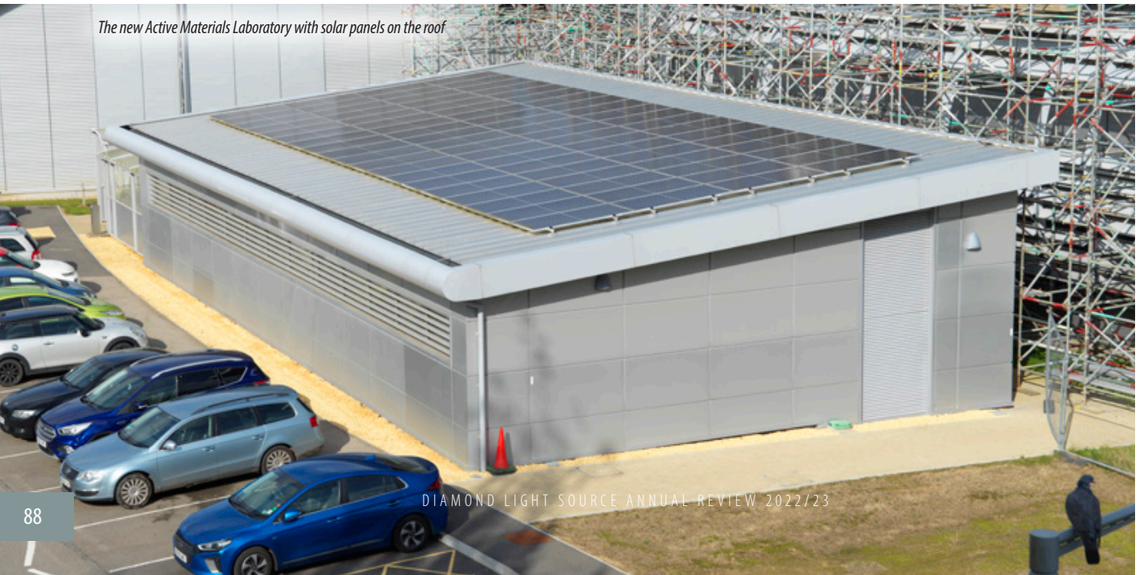
The strategy divides environmental sustainability into three areas; Research, operations and compliance.

Research Impact: These are areas where Diamond is having a positive impact on sustainability through the research and technology development that we facilitate. Our strategy is to maximise the positive impact of these areas. Our Diamond-II infrastructure upgrade programmes has enhancements to these positive impacts at the centre, with the primary commitment of the strategy focusing on directly addressing the Government's Industrial Strategy Grand Challenges of clean growth, mobility and an ageing society.

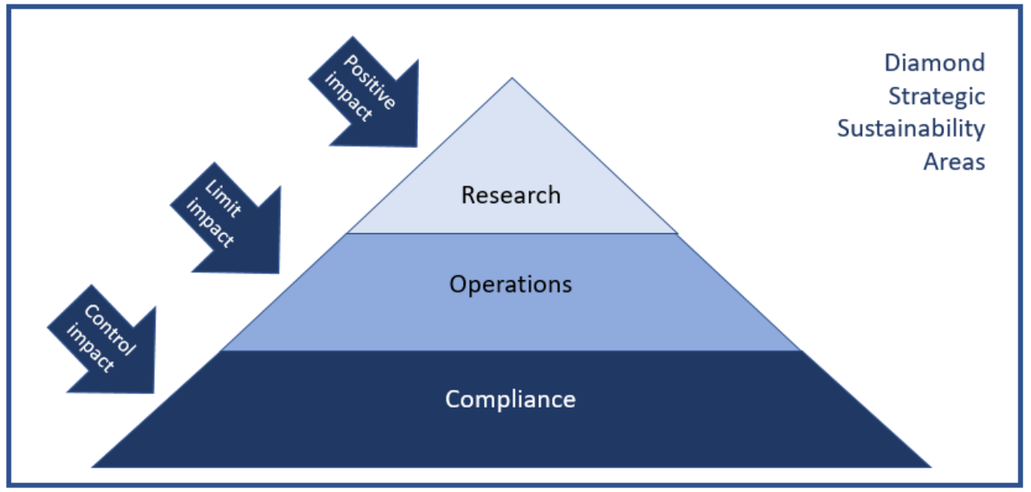
This also includes addressing climate change challenges.

Sustainability research areas at Diamond include:

- **New batteries**: to reduce the carbon footprint linked to the exploitation of batteries and particularly batteries involving rare earth materials, researchers are working on all the elements of a battery: electrolytes, anode, cathode, interface of all the elements. The goal is to limit or even replace the usage of lithium with safer, and more abundant materials (Sodium for example).
- **Hydrogen production:** To reduce the usage of fossil fuel and limit the production of CO_2 or other atmospheric pollution, new ways to produce Hydrogen are being developed by researchers. This will allow the production of H_2 more efficiently, with less cost involved (photocatalysis, or usage of bioreactors with a neutral cost).
- **Photovoltaic development:** The sun is a limitless source of energy for our societies. Nevertheless, researchers are searching new ways to improve the energy production from photovoltaic panels, including new materials (Perovskites or other materials) or increasing the efficiency of existing materials (with an increased absorption window).
- **Plastic depollution:** Plastic pollution has dramatic consequences for the environment. Researchers are working on new enzymes (PETase) to degrade microplastic, reducing plastic pollution and allowing the production of valuable molecules at the same time.
- **Atmospheric pollution:** Research has been conducted at Diamond to understand the ageing and decay of aerosols produced by human activities such as cooking, a source of pollution in large cities.
- **Radioactive pollution:** Different events such as nuclear testing or Reactor meltdown (Fukushima -Daichi, for example) has caused major environmental pollution by radioactive elements. Diamond beamlines are used to understand how the soil is reacting to radioactive pollution and what can be the short- and long-term impact to such pollution.
- **Ecosystem evolution:** Fragile environments such as coral reefs are modified following direct or indirect human activities Synchrotron studies help to understand the evolution of such environments, and how to protect them.
- **Pollution remediation:** Human activities can produce local or global pollution, that can be detrimental for the environment. Researchers are developing new materials to remove toxic elements such as Arsenic or sulphur; or characterising already existing organisms such as algae to understand how they can accumulate toxic elements (Cadmium, for example)
- **Green chemistry:** Scientists are working on new enzymes to enhance the production of valuable molecules with a lower environmental cost. In a comparable manner, enzymes or other materials such as Metal-Organic Framework are modified to process abundant molecules resulting from human activities such as lignin or CO_2 into useful compounds.



The new Active Materials Laboratory with solar panels on the roof



Operational Impact: These are areas related to the operation of Diamond that have impacts on environmental sustainability, such as energy usage. Our strategy is to limit the impact of these areas through a commitment to continual improvement of our performance. Our approach to continuous improvement will be underpinned by adoption of best practice, regular review, and evaluation, monitoring of progress and the identification of areas for development.

Through investment in a wide range of energy saving measures, such as variable speed drives on pumping equipment, motion sensors on lighting and LED light bulbs, Diamond has already achieved ongoing electricity savings of over £1million per annum and continues to identify areas of improvement. The primary focus for energy and resource usage is to work towards 'net-zero' carbon emissions for our directly managed operations by 2040.

Other primary focus areas relating to operational impact include working towards an ambition of zero avoidable waste by 2050; working towards eliminating all avoidable plastic waste by 2042, with earliest possible elimination dates as alternatives and technologies become feasible; developing a procurement culture which prompts staff to consider environmental responsibility and sustainability in their purchasing decisions; and developing sustainable design policies contributing to an environmentally sustainable facility, for the present and future.

The key commitments of our strategy for sustainable operations include the following:

Decision Making & Engagement: We will embed environmental sustainability objectives into Diamond Executive's objectives and business plans.

Energy Resource and Usage: We will perform annual reviews of energy usage metrics, purchasing options and travel related carbon emissions to set reduction targets and select the greenest viable supply sources.

Waste: We will minimise waste, reuse materials as much as we can and manage materials at the end of their life to minimise the impact on the environment.

Sustainable Design: We will optimise the leadership contribution of sustainable design to contribute towards an environmentally sustainable facility, for the present and the future.

Compliance Impact: These are areas where environmental sustainability requirements are mandated by legislation or guidance, for example, environmental permits and authorisations. Our strategy is to continue to control the impact from these areas through our management system and robust procedures and processes.

Related publications:

Wang, X. *et al.* Atomically dispersed pentacoordinated-zirconium catalyst with axial oxygen ligand for oxygen reduction reaction *Angew. Chem. Int. Ed.* **61**, e202209746. (2022) DOI: 10.1002/anie.202209746

Potter, M. *et al*. Combining photocatalysis and optical fiber Technology toward improved microreactor design for hydrogen generation with metallic nanoparticles. *ACS Photonics* **7**, 3 (2020) DOI: 10.1021/acsphotonics.9b01577

Taddei, M. *et al.* Ethylenediamine addition improves performance and suppresses phase instabilities in mixed-halide perovskites. *ACS Energy Lett.* **7**, 12, (2022) DOI: 10.1021/acsenergylett.2c01998.

Erickson, E. *et al.* Sourcing thermotolerant poly(ethylene terephthalate) hydrolase scaffolds from natural diversity. *Nat Commun.* **13**, 7850 (2022). DOI: 10.1038/s41467-022-35237-x

Milsom, A. *et al.* The impact of molecular self-organisation on the atmospheric fate of a cooking aerosol proxy, *Atmos. Chem. Phys.* **22**, 4895–4907, DOI: 10.5194/acp-22-4895-2022, 2022.

Cook, M. *et al.* The nature of Pu-bearing particles from the Maralinga nuclear testing site, Australia. *Sci Rep* **11**, 10698 (2021). DOI: 10.1038/s41598-021-89757-5

Smith, G.L. *et al.* Reversible coordinative binding and separation of sulfur dioxide in a robust metal–organic framework with open copper sites. *Nat. Mater.* **18**, 1358–1365 (2019). DOI: 10.1038/s41563-019-0495-0

Thorpe, T.W. *et al.* Multifunctional biocatalyst for conjugate reduction and reductive amination. *Nature* **604**, 86–91 (2022). DOI: 10.1038/s41586-022-04458-x

Industrial Liaison

Industrial Liaison Office

Following on from the upheaval of the pandemic years, 2022 felt like a return to the stability of former times. As the world settles into the new normal way of working, our years of preparation in developing remote access, mail in data collection and a range of services for our clients enable us to react quickly to our clients' changing needs. The industrial programme at Diamond continues to thrive with fully operational life and physical science programmes. Building on last year's successes, we are again celebrating our busiest year to date.

The increase in industrial engagement has also naturally led to expansion of the team and this year we welcome Isabel Barker to the industrial XChem team as a Research Technician. Izzy recently achieved her first class MSci (Hons) degree in Biochemistry at the University of Sussex and is bringing her skills in protein preparation and macromolecular crystallography to complement the XChem team and support delivery of industrial XChem campaigns.

With travel becoming possible once again, our clients returned to Diamond to perform a wide range of experiments and our team were able to travel and attended conferences and events, meeting clients and friends both old and new. Highlights include members of the life sciences team attending Protein Structure Determination in Industry (PSDI) in Eindhoven and the Novalix conference in Munich along with the Fragments and MiBio conferences both taking place in Cambridge. Representing the physical science team, Anna Kroner gave an invited talk at the XAFS 2022 conference in Sydney, Australia. Rachel Freeman represented Diamond at the Big Science Business Forum in Granada, working with counterparts at other European research infrastructures to engage with industry. We are now looking forward to meeting our valued clients at wide range of events over the coming year.

Diamond continues to support our industrial clients across a wide range of techniques and facilities with experiments ranging from routine analysis to complex bespoke solutions. Please do not hesitate to contact us on industry@diamond.ac.uk to discuss your analytical needs, we'd be happy to help.

Alex Dias and Ailsa Powell preparing pucks for XChem experiments.

Engaging with Diamond Light Source

Communications and Engagement Team

Over the past year the impact of COVID pandemic restrictions on Diamond's engagement activities has lessened and in person activities have resumed to pre-pandemic levels. The knowledge and experience we acquired in developing and delivering remote activities have not been abandoned, as virtual activities have been integrated into many of our existing programmes from events to school engagement, maintaining the increased accessibility this format allows.

During the 2022-23 period, Diamond has had approximately 8098 significant interactions[i] with visitors, an increase from the previous year (7323 interactions). These include 2890 for scientific and technical events, 452 undergraduate and postgraduate interactions, 4364 school students and members of the public, and 392 VIPs and Stakeholders. In 2022, the majority of these interactions were virtual, however in 2023 we have seen a move back towards in person events, with the majority happening in person onsite (3594) or in person offsite (1249). While most interactions have happened in person, we are proud to have maintained a high level of interaction with visitors online (3255), increasing the accessibility of Diamond to a wide range of audiences.

[i] Significant Interaction is defined as a talk/tour/meeting of 30+ minutes, typically longer.

In 2023, we have worked hard to transition from an almost exclusively remote program of activities put in place to continue to engage audiences during the pandemic, back to in person activities once again. We successfully reinstated our core program of in-person Public Engagement activities, but expanded this to include successful virtual activities that reached underserved audiences during the pandemic. For example, we have continued to run bespoke virtual school visits, whereby individual schools can request a virtual visit to Diamond to fit around their school timetable. This continues to give access to Diamond to all schools, regardless of their situation. We have continued to work with our existing partners such as STFC, at RAL and Daresbury, and Canterbury Christ Church University, to reach both new and larger audiences.

> *"We loved the experience and found it absolutely fascinating. Thank you to the whole team for making us welcome, showing us around and, in particular, inspiring the next generation."*

Public Inside Diamond open day attendee, March 2023.

In July 2022 we were delighted to be able to run a fully in person Schools/College Work Experience week for students in years 10-13. We welcomed 48 students across 24 projects, a record-breaking number of both students and projects for Diamond's Work Experience Academy program. Projects spanned all divisions of Diamond, from mechanical engineering to electron structure. On the student's final day at Diamond we welcomed MP David Johnston to help celebrate the success of the programme.

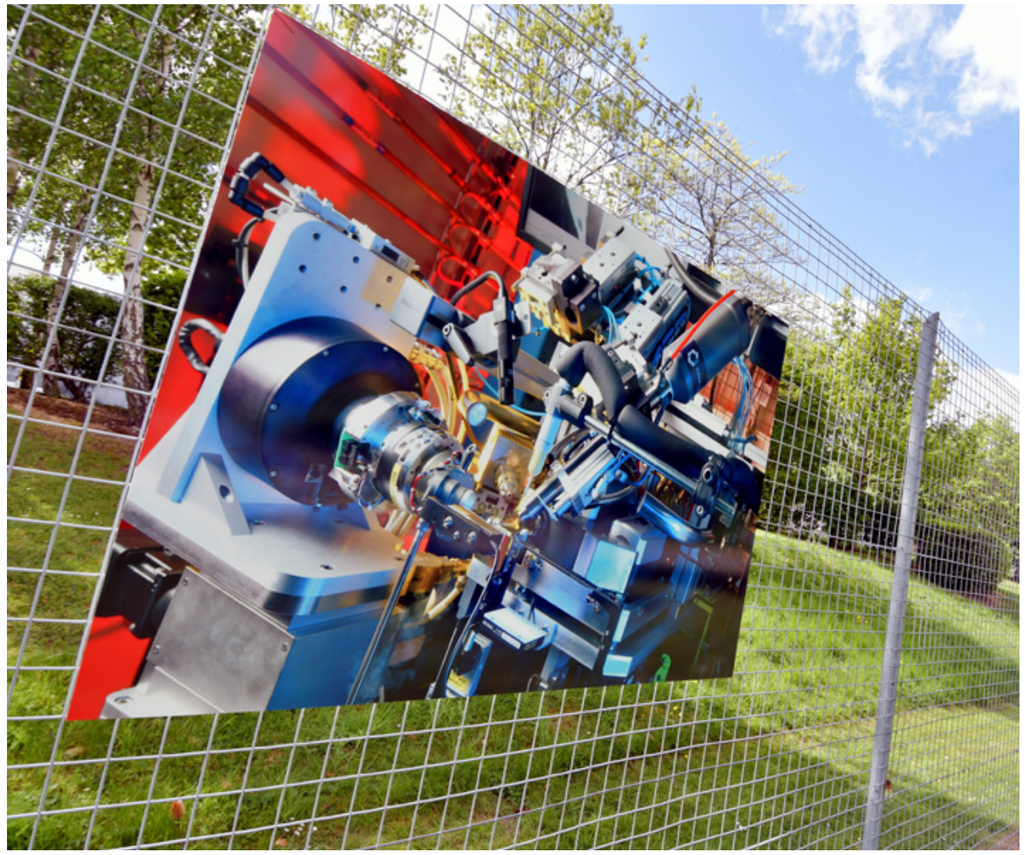
> *"It was so exciting to work at the facility and see the synchrotron in real life and to learn about the multitude of research projects being undertaken! It was a real privilege to work on the nanoimaging of Li-ion batteries project with Dorota as my supervisor and the placement has further sparked my interest in Materials Science and Engineering."*

Work Experience student 2022.

In addition to the annual Schools/College Work Experience week, we also ran a special one-off project with a small group of students trailing the functionality and capabilities of Diamond's Visible Synchrotron Radiation (ViSR) beamline, a resource developed to allow visitors to interact with visible synchrotron light and carry out simple experiments. The students joined us for 3 days and worked together to ensure ViSR is fully functional and ready to be used for future engagement activities.

> *"The project opened my eyes to how science as a whole is conducted. It was fascinating to learn exactly what happens in and around Diamond."*
>
> *"It was a good way to visualise areas we learn in school and it is nice to see theory applied in the real world."*

Students taking part in the ViSR Project.



Diamond has continued its commitment to widening participation and dedicated more efforts towards community engagement, working with groups such as the Amos Bursary, Science Oxford, Science Together, the Careers Transition Partnership and the Social Mobility Foundation, delivering/supporting outreach activities in a variety of formats to audiences with lower science engagement.

In 2022 Diamond celebrated its 20th anniversary and 15 years of research and innovation, and to help commemorate the occasion we presented to the public a special exhibition of selected photographs from throughout Diamond in an outdoor photographic exhibition. The images circumnavigated the "ring" of the synchrotron along the perimeter fence and gave a behind-the-scenes insight into that goes in inside our iconic building. The exhibition formed part of the wider Oxfordshire Artweeks festival, highlighting our work to the community in an accessible format.

Higher Education Engagement

Our work with students at both undergraduate and postgraduate level continues to play a vital role in Diamond's wider mission to be a world-leading centre for synchrotron science and to keep the UK at the forefront of scientific research. Our student-dedicated programmes aim to welcome and harness the talent, curiosity and development of students both at undergraduate and postgraduate level and provide them with exciting opportunities to encourage and nurture a career in STEM, ultimately contributing to the wider skills agenda in the UK.

In 2022, 23 joint-PhD students joined Diamond as part of our 2022 PhD cohort. These doctoral projects are linked with 16 universities and other world leading facilities, and we have been able to welcome the majority of them on an individual basis here at Diamond. This brings the total number of active Diamond PhD Studentships to 109. We received 62 submissions for our 2023 Diamond Doctoral Studentship call for proposals, which were linked with 32 different universities and institutions. Following the internal review process, we will be welcoming 26 students in October 2023.

This year, we held a PhD Development Days Event, open to all our jointly funded students. It was a 2-day event with a networking dinner on the first day. Day 1 was career focused with talks from a range of different areas from those still in the scientific field through to publishing, patents and government. We were thrilled to have three Diamond PhD alumni talk as part of the careers panel. Day 2 focused on personal resilience development for the PhD students, aiming to engage students to step back, train their personal resilience and start a process of identifying their professional purpose. We had 31 PhD students attending the event overall.

A student commented:

> *"I very much enjoyed the Development Days, not only was it useful to hear talks from people from different careers, it was also valuable to be able to network and chat with other students in the evenings."*

The 2021 Year in Industry cohort finished their placements in September, and we were able to have their final poster session and presentations in person,

giving wider Diamond staff the opportunity to gain insight into their research, any challenges and results during their 12-month placement.

"Doing a year in industry at Diamond Light Source provides a life-changing opportunity for any undergraduate student, and a massive stepping stone onto the career ladder. This paired with an amazingly supportive student engagement team, and a strong community between the year in industry students makes the 12 months that you'll spend here fly by!"

We were pleased to welcome 12 new Year in Industry students as part of the 2022 cohort intake in September and to have them in-person and on-site at Diamond. Since starting the 2022 cohort have been busy progressing their projects, completing various training including Scientific Communications, Peer Reviewing and Presentation Skills as well as getting involved with wider outreach activities.

In June 2022, we welcomed our 12 Summer Placement students. As restrictions had been fully lifted, we were delighted to have the cohort working fully in-person and on-site at Diamond. The projects spanned life and physical sciences, engineering and software computing. After a summer of work experience, training and networking, the students presented their results to staff via a poster event in the Atrium and end of project presentations.

Due to the lifting of COVID restrictions, Diamond was once again able to welcome undergraduate and postgraduate students in person to Diamond, where we offered a range of talks and training. We welcomed 452 students for this type of visit, which is comparable to pre-pandemic levels.

Scientific workshops and conferences

Diamond organises a broad portfolio of scientific and technical workshops, training courses and conferences tailored towards the needs of our staff and user communities. In 2022, we hosted 38 events and engaged with 2,890 scientists and engineers from all over the globe.

In August 2022, Diamond hosted the 12th International Conference on Inelastic X-ray Scattering (IXS2022). The conference was held at the SAID Business School in the city of Oxford. Taking place over five days, we welcomed international experimentalists and theorists interested in the use and development of both Resonant and Non-resonant Inelastic X-ray Scattering to address a broad range of materials science challenges.

In the autumn, we hosted the 6th edition of our Early Career Scientists Symposium. This annual event featured talks from eminent speakers such as Nobel Prize winning biochemist Prof. Jennifer Doudna and theoretical physicist, author and broadcaster Prof. Jim Al-Khalili. The symposium was by far the most popular event of the last 12 months and brought together over 800 junior scientists looking for inspirations to shape their future careers.

Amongst many workshops and user training opportunities, we moved into 2023 with events focused on boosting national and international collaborations. These included the annual Lightsources.org event, which focussed on the capabilities and career opportunities at 4th generation light sources, and the CONEXS Conference, which highlighted x-ray spectroscopy to achieve new levels of understanding, especially for the interpretation of experimental data.

With an eye on the future and the prospect of the Diamond-II upgrade on the horizon, we also ran a series of scientific workshops to directly engage with our academic and industrial user communities to explore how Diamond-II can best support research and innovation.

Our 2022 Year in Industry Cohort after completing their two-day Presentation Skills Workshop, held at Cosener's House in Abingdon, March 2023.

CONEXS 2023

Governance and Management

Diamond Light Source Ltd was established in 2002 as a joint venture limited company funded by the UK Government via the Science and Technology Facilities Council (STFC), now under UK Research & Innovation (UKRI), and by the Wellcome Trust, owning 86% and 14% of the shares respectively. Diamond now employs 814 scientists, engineers, technicians and support staff from 44 countries worldwide. The Chief Executive and Directors are advised by committees representing key stakeholder groups, including the Science Advisory Committee (SAC), Diamond Industrial Science Committe (DISCo) and Diamond User Committee (DUC).

Diamond is free at the point of access for researchers accessing Diamond via peer review, and provided the results are published in the public domain for everyone's benefit. Allocation of beamtime is via a peer review process to select proposals on the basis of scientific merit and technical feasibility. Twelve peer review panels meet twice a year to assess the proposals submitted for each six-month allocation period. Diamond also welcomes industrial researchers through a range of access modes including proprietary research.

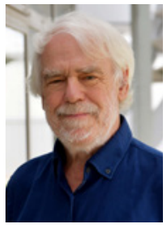
Board of Directors

Prof. Sir Adrian Smith (Chairman)
Marshall Davies
Prof. Michael Fitzpatrick
Dr Morag Foreman
Prof. Mark Thomson
Andrea Ward
Prof. Keith Wilson

Company Secretary
Linda Gregory

As at April 2023

Executive



Prof. Sir David Stuart is MRC Professor of Structural Biology at the University of Oxford, and Joint Head of the Division of Structural Biology at the Department of Clinical Medicine. He was appointed Director of Life Sciences at Diamond in 2008. His principal research interests include the structure of viruses and viral proteins as well as cellular proteins, especially those that interact with viruses. Dave was knighted in the New Year Honours list 2021 for services to medical research and the scientific community.

Prof. Richard Walker joined Diamond Light Source as Technical Director in January 2002. He was previously Director of the Light Sources Division at Sincrotrone Trieste in Italy, and prior to that he was a key member of the Daresbury Laboratory SRS team. Richard is a visiting Professor of Physics at the University of Oxford.

Andrea Ward joined Diamond Light Source in 2019, with 15 years experience as a Senior Finance professional, and is CEO and Director of Finance and Corporate Services. During a 12-year tenure at Vertex Pharmaceuticals, she worked with the Board to lead finance and procurement functions in Europe, later moving to Canada with the business to assist with acquisition and commercialisation opportunities. Andrea has also worked at ResMed and the Ontario Lottery and Gaming corporation.

Dr Adrian Mancuso joined Diamond in November 2022 from the European XFEL facility, where he has held the position of Group Leader and Leading Scientist for the Single Particles, Clusters, and Biomolecules & Serial Femtosecond Crystallography (SPB/SFX) instrument Group since 2010. As Physical Science Director, Adrian is responsible for overall strategic leadership, direction and management of Diamond's Physical Science Division.

Staffing and Financial Information

Outline Organisational Chart

Chief Executive's Office
Communications, Engagement & Impact
Industrial Liaison
Safety, Health & Environment

Science Division

Science Groups:
Biological Cryo-Imaging
Crystallography
Imaging and Microscopy
Macromolecular Crystallography
Magnetic Materials
Soft Condensed Matter
Spectroscopy
Structures and Surfaces

Scientific Software, Controls & Computation Groups:
Accelerator Control Systems
Beamline Control Systems
Data Acquisition
Scientific Computing
Scientific Software

Detector Group
Experimental Hall Labs Services
Optics & Metrology
Planning & Projects Office
User Office

Technical Division
Accelerator Physics
Diagnostics
Engineering
Insertion Devices
Installation & Facility Management
Operations
Power Supplies
Radiofrequency Systems
Vacuum

Finance and Corporate Services
Corporate IT
Commercial Management, Governance & Legal
Finance
Human Resources
Procurement & Goods Handling
Soft Facilities

Summary of Financial Data

	2013/14	2014/15	2015/16	2016/17	2017/18	2018/19	2019/20	2020/21	2021/22	2022/23
Operating Costs £m	42.5	44.5	54.6	56.9	62.8	64.5	65.7	69.2	68.7	76
Total Staff (Year End)	507	534	582	609	639	680	742	775	786	814
Capital Expenditure – Operations £m	7.5	6.2	8.0	10.5	12.8	17.4	17.8	24.1	21.2	22.3
Phase II £m	0.8	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Phase III £m	17.2	23.7	20.6	11.5	3.7	1.0	0.0	0.0	0.0	0.0
Other capital projects £m		4.8	5.6	7.3	4.3	5.3	1	2.1	2	3.7

Figures up to and including 2013/14 exclude VAT, thereafter figures include VAT.

Committee Membership

The Scientific Advisory Committee (SAC) advises the CEO and the Science Directors on the scientific and technical questions impacting the specification, design, commissioning and operation of the facility; experimental and user support facilities, and opportunities for scientific exploitation.

Dr Tom Hase (Chair)
University of Warwick (UK)

Dr Paul Adams
Lawrence Berkeley National Laboratory (USA)

Dr Elke Arenholz
Pacific Northwest National Laboratory, USA

Prof Rohit Bhargava
University of Illinois Urbana-Champaign (USA)

Dr Dina Carbone
MAX IV (Sweden)

Prof Kristina Djinović-Carugo
EMBL Grenoble

Prof Peter Dowding
Infineum (DISCo Representative)

Prof Chris Hardacre
University of Manchester (UK)

Prof Phil King
University of St Andrew (UK)

Prof Matt Rosseinsky
University of Liverpool (UK)

Prof Andrea Russell
University of Southampton (UK) - (Chair of the DUC)

Prof Christian Schroer
DESY (Germany)

Prof Stephen Skinner
Imperial College London (UK)

Prof Xiaodong Zhang
Imperial College London (UK)

Prof Elizabeth Wright
University of Wisconsin-Madison (USA)

Membership as of May 2023

The Diamond Industrial Science Committee (DISCo) advises the CEO and Directors on opportunities for industry to be engaged in research at Diamond, industrial research priorities that will help shape operational strategy, including the best way to exploit the current suite of beamlines and to develop the case for investment in future beamlines, and to develop best practice for industrial engagement.

Dr Malcolm Skingle
GlaxoSmithKline (Chair)

Dr Andrew Barrow
Rolls-Royce

Dr Cheryl Doherty
GlaxoSmithKline

Dr Helen Blade
AstraZeneca

Dr Rob Cooke
Sosei Heptares

Prof. Peter Dowding
Infineum

Prof. Jonathan Hyde
NNL

Dr Olga Kazakova
NPL

Dr Ellen Norman
RSSL

Dr Pamela Williams
Astex Pharmaceuticals

The Diamond User Committee (DUC) has been set as a platform for discussion between Diamond and the user community of matters relating to the operation and strategy of Diamond.

Dr Imad Ahmed
University of Oxford

Dr Arnaud Basle
University of Newcastle

Dr Gavin Bell
University of Warwick

Dr Jamie Blaza
The University of York

Dr David Briggs
The Francis Crick Institute

Dr Ann Chippindale
University of Reading

Dr Sean Connell
CIC bioGUNE

Dr Kevin Edmonds
The University of Nottingham

Dr Enrique Jimenez-Melero
The University of Manchester

Dr Tim Knowles
University of Birmingham

Dr Marcus Newton
University of Southampton

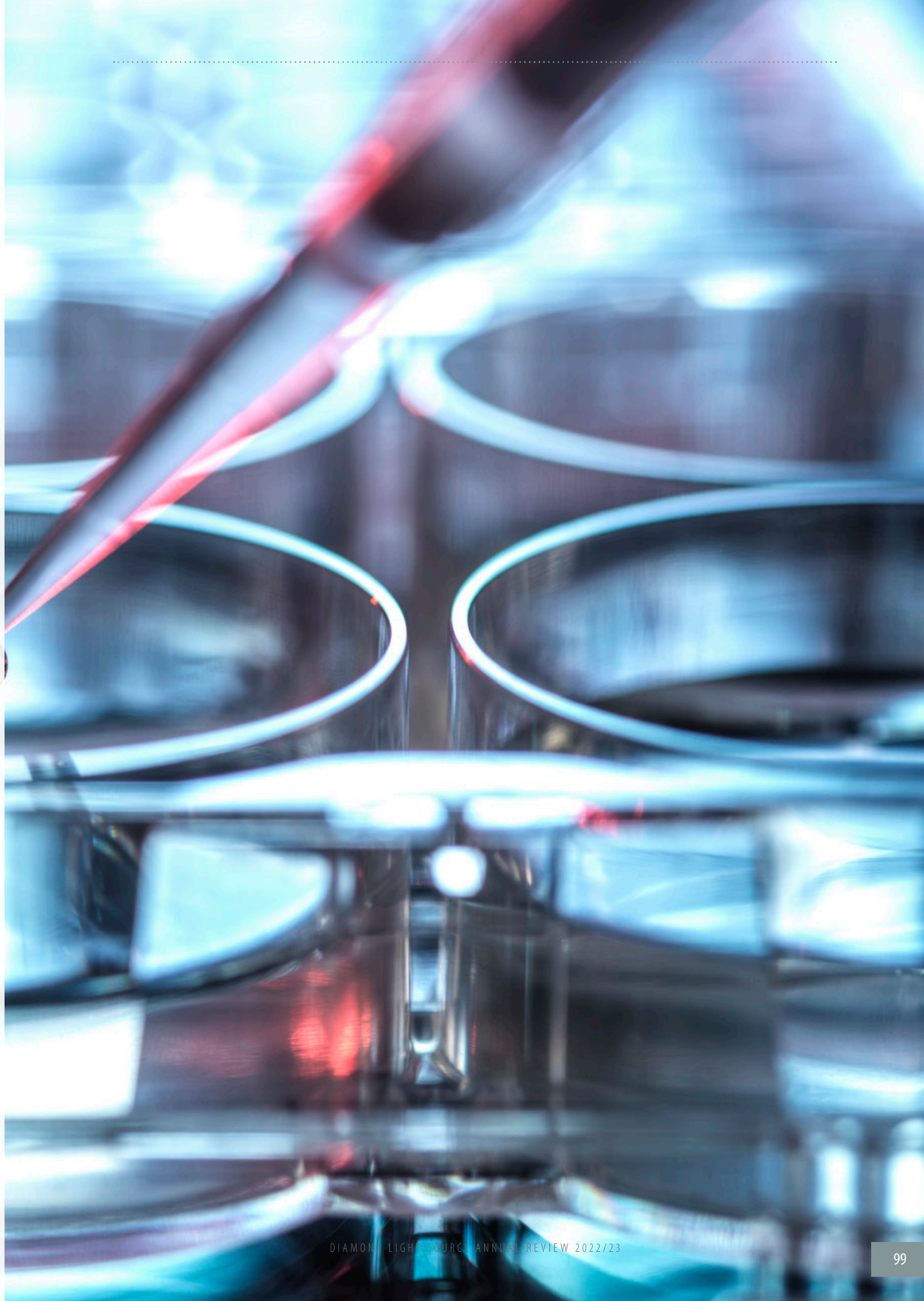
Dr Robin Perry
University College London

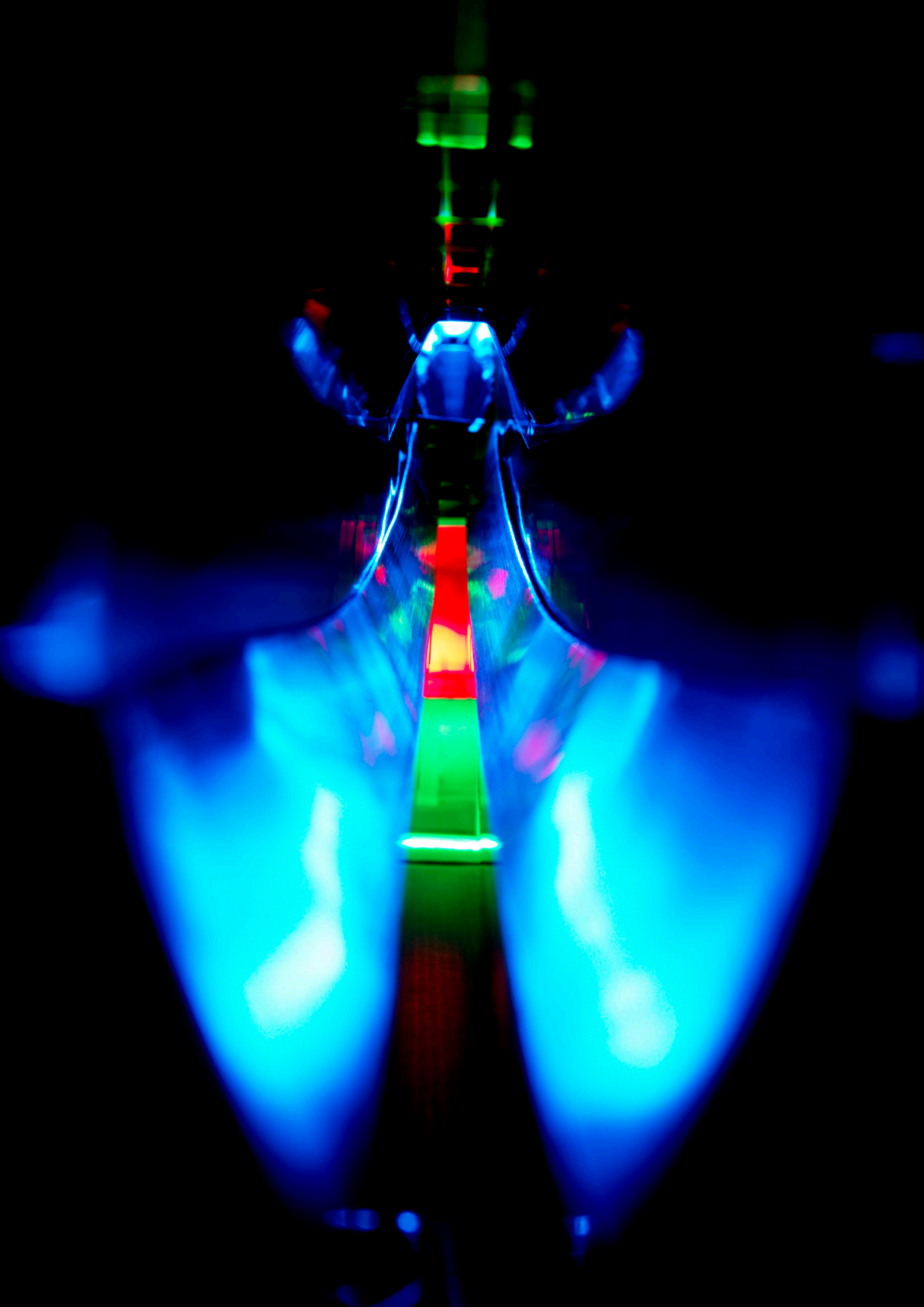
Prof Andrea Russell
University of Southampton (Chair)

Dr Neil Telling
Keele University

Dr Andrew Thomas
The University of Manchester

Dr Arwen Tyler
University of Leeds





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