Diamond Light Source Ltd Review 2017/18



Mechanical Beamline Technician, Peter Garland, working on the sample station in the extensive experimental hall of 121.

Foreword

Professor Sir Adrian Smith *Chairman of the Board of Directors*

Diamond is one of the world's leading synchrotrons, providing its industrial and academic user communities with access to state-of-the-art analytical tools.

The resulting knowledge has wide-ranging impacts - scientific, economic, cultural and societal. The many high-impact peer-review publications we deliver attest to our scientific excellence. Equally important is the breadth and depth of engagement with industry and the resulting translation into economic impact, evidenced by over 125 companies paying for proprietorial access and an increasing number of companies engaging through partnerships with the many university groups who access Diamond. As we look to the future, the key challenge ahead will be to secure the investments in the facility that will ensure our continuing international leadership position. The Diamond Board will continue to use its influence to ensure that we maintain and enhance our leading global reputation.

The many high-impact peer-review publications we deliver attest to our scientific excellence.

CEO Welcome

marked a double anniversary for Diamond: our 15th anniversary as a company and 10 years of research and innovation. This was marked by the official visit of HRH the Princess Royal last February - a tremendous accolade for everyone involved with Diamond.

Our facility is currently completing its final beamlines, which will see us close Phase III construction in 2019. We now have 31 operational beamlines which are delivering an increasing number of high-impact publications. This review presents some of the highlights of this science, illustrating both new insights we are providing in fundamental science, and the positive impact for the economy and society at large in partnership with our user community.

With over 9,000 user visits, a machine up time of 98.2%, and exceptionally dedicated staff supporting every aspect of our activities, our offer to the science community is second to none. All of this underpins outputs that grow from year to year, most readily illustrated by the number of peer-reviewed publications, numbering 6,825 in 2017 with 41% of the output linked to the life sciences. This is the result of our creating an integrated offer for our structural biology community, which is unique in the world. We now have six microscopes dedicated to the life sciences running seven days a week each delivering 220 days of science per year. We are becoming a one-stop

shop to understand the origins of diseases at a molecular or cellular level and to both discover and develop drugs to treat them. The physical sciences are blooming too, from multiprobe and operando studies of catalysts to new emerging technologies for ICT such as multiferroics, where electrical and magnetic functionality are coupled, so changing one can switch the other. As you will read in our Materials and Magnetism section, researchers from across the globe have used our IO6 beamline to visualise this phenomenon and as the basis for new multifunctional devices that work well at room temperature, are opening up new avenues for creating new electronic storage devices. Engaging with industry is also an essential part of our mission and the past year has seen a record income from proprietorial access. An increasingly important part of this is the new embedded facilities for electron microscopy for both life and physical sciences at eBIC and ePSIC, respectively. Diamond also provides crucial facilities for metrology, illustrated recently by work performed by FMB Oxford to test the performance of their manufactured components.



As taxpayers essentially fund 86% of the facility, we have kept our commitment to engage with the public. We offer regular open days and work with the media to communicate and create greater awareness of the ground-breaking science achieved here. With 5,635 visitors in the past year alone, of which 3,121 were school students and members of the public, we are continually making sure we present strong and diverse role models across all disciplines. We want to inspire as many youngsters as possible by the work we do and persuade as many of them as possible to choose STEM subjects as the path to their future careers.

If we are to continue to enable world-leading science and innovation, we must ensure that Diamond continues to offer the very best technical facilities, from the machine, through the beamlines to rapid and effective methods of data analysis. In recognition of this, our stakeholders - STFC and the Wellcome Trust - will increase our capital budget from 2019 to support an ambitious programme of rolling upgrades. We prioritised these upgrades in close consultation with our advisory bodies for academia and industry, as well as reflecting on the UK Government's Industrial Strategy, which presented grand challenges to which we can strongly contribute - clean growth, mobility and an ageing society. Our own plans also resonate with wider developments across the Harwell Campus – in particular the Rosalind Franklin Institute to develop enabling technology for medicine and the



Faraday Institution for battery research – in which we are to be key partners. In the longer term, we plan a wholesale upgrade of machine itself – Diamond-II – in parallel with machine upgrades at synchrotrons around the world. This will offer an increase in brilliance, coherence and capacity that will offer wholly new scientific opportunities and maintain our competitive edge.

However, such developments should be carried out in partnership with other synchrotrons, which share the same technical challenges and aim to solve similar scientific problems. To this end, Diamond has played an active role in founding the League of Accelerator-based Photon Science (LEAPS), which brings together 16 organisations across Europe representing all 19 European synchrotrons and free electron laser facilities. As Brexit approaches in March 2019, it will be critical to ensure the UK remains a key element of this network and we continue to benefit from the international cooperation and exchange with our strongest international partners that enable us to tackle together some of the key scientific and societal challenges of our time.

Prof Andrew Harrison

CEO Diamond Light Source

Governance

iamond 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), and by the Wellcome Trust, owning 86% and 14% of the shares respectively. Diamond now employs over 630 scientists, engineers, technicians and support staff from over 40 countries worldwide. The Chief Executive and Directors are advised by committees representing key stakeholder groups, including the Science Advisory Committee, Diamond Users' Committee, and Diamond Industrial Science Committee.

Diamond is free at the point of access for researchers, provided the results are put in the public domain. Allocation of beam time is via a peer-review process to select proposals on the basis of scientific merit and technical feasibility. Eight 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.



Senior Beamline Scientist, Phil Chater loads a sample on 115-1.



M-CXL95-50

Instruments



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
Talos Arctica	Cryo-EM, cryo-ET	200 kV	Operational since 2016
Scios	Cryo-SEM, Cryo-FIB	3 to 30 kV	Operational since 2017
JEOL ARM200F	EDX, EELS, atomic scale STEM imaging, electron diffraction	80, 200 kV	Operational since 2017
JEOL ARM300F	EDX, atomic scale TEM and STEM imaging, electron diffraction	30, 60, 80, 160, 200, 300 kV	Operational since 2017



Key Facts and Figures

Facility usage

In our eleventh year of operations (1st April 2017 to 31st March 2018), we received 1,515 proposals for experiments on our instruments via peer reviewed access routes, requesting a total of 19,507 shifts. After peer review 1,095 proposals were awarded beamtime. This resulted in 11,667 experimental shifts being awarded across 28 operational beamlines, one commissioning beamline and eight electron microscopes. We welcomed 5,668 onsite user visits from academia across all instruments, with an additional 3,656 remote user visits.

In the last 12 months Diamond started to review its reporting method on facility usage. A new reporting tool is currently being developed and allows us to report more precisely and more consistently. As a result, some of the reporting criteria in this review have evolved compared to previous years. Further efforts on reporting are being carried out and should continue in the next 12 months.

User shifts requested, awarded and delivered by group, beamline and electron microscope 2017/18



Requested Awarded Delivered

* B21: Until April 2018, some B21 shifts are requested as part of MX BAG proposals. These shifts are not included as requested shifts in the graph above.

• IO4-1: some delivered shifts are delivered as part of XChem campaigns which are not included in this report

• For I11, LDE experiments are not included in this section.

• 113-2 Provides 35% of the available shifts to the Diamond Manchester Collaboration: these shifts are not included in this section.

B16 is an optical testing beamline with only 50% of beamtime for users

Total user shifts requested, awarded and delivered



📕 Requested 📕 Awarded 📕 Delivered

Total numbers of proposals and users per year



Proposals submitted Proposals awarded User visits Individual remote user visits

Proposals by discipline and research theme

Experimental shifts scheduled by Diamond by main subject area for 2017/18



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



Machine performance

	2008/9	2009/10	2010/11	2011/12	2012/13	2013/14	2014/15	2015/16	2016/17	2017/18
Total no. operational beamlines by end FY	13	17	19	20	22	24	25	26	28	28
Scheduled hours of machine operation	5640	5712	5808	6000	5832	5976	5808	5928	5688	6072
Scheduled hours of user operation	4305	4728	4728	5064	4872	5088	4944	5040	4584	5160
Machine uptime %	95.0	97.0	97.5	97.7	98.3	98.2	97.6	97.6	98.7	98.2
Mean time between failures (hours)	15.3	26.2	28.5	55.4	52.4	60.3	38.6	119.4	103.1	79.9

* no posters

Science Highlights

Macromolecular Crystallography

acromolecular Crystallography (MX) reveals the shape and arrangement of biological molecules at atomic resolution, providing a highly accurate insight into their function. Users can combine MX with the many other techniques available at Diamond, and lab-based investigations, to reveal the broader picture of molecular interactions and their effects.

The MX group is recognised as an innovative world leader, moving the goalposts of what is feasible for 'conventional' MX and developing techniques and beamlines that enable new experiments and methodologies.

In 2017, studies included those identifying potential new treatments for diabetes, investigating one mechanism behind the spread of antibiotic resistance, and gaining key insights into a glycoprotein essential for healthy heart development and blood vessel growth.

Identifying potential treatments for diabetes

Diabetes and obesity are serious public health concerns, putting great pressure on healthcare systems across the globe. Developing effective and affordable drugs to treat these conditions remains a pressing need.

Glucagon-like peptide 1 (GLP-1) is an important hormone released from the gastrointestinal tract in response to food intake. GLP-1 promotes the release of insulin from the pancreas to regulate blood sugar, through its interaction with its binding partner, the glucagon-like peptide 1 receptor (GLP1R).

The stimulation of GLP1R is considered an attractive means of treating type 2 diabetes but although there are several medically-approved GLP-1 peptide analogues, these are expensive to produce and must be administered by injection. Alternatives that could be taken orally would significantly simplify the treatment regimen for people with type 2 diabetes.

Researchers used the MX Microfocus beamline (I24) to produce a detailed map of the interactions between GLP-1 and its receptor, which enabled the design of smaller GLP-1 analogues that were active in a mouse model of diabetes. These new analogues could be the precursors of future diabetes treatments.

Jazayeri A et al. doi: 10.1038/nature22800

Tackling antibiotic resistance

Bacterial diseases, particularly those caused by Gram-negative bacteria (GNB), are a serious and increasing clinical threat worldwide. Treatment of GNB infections is complicated by the spread of antibiotic resistance, and there are now some bacteria for which few treatment options remain.

Colistin is a key `last-resort' antibiotic, used to treat infections by multidrugresistant GNB pathogens. Until recently, resistance to colistin was considered rare, but in November 2015 research described colistin resistance mediated by MCR-1, a plasmid-encoded phosphoethanolamine (PEA) transferase. It has now been reported in over 40 countries, and is found in numerous bacterial strains, including *E. coli*.

To understand resistance to Colistin, researchers have used beamlines I02, I03 and I04-1 to obtain the crystal structures of the catalytic domain of the transferase MCR-1. These data, alongside their recently determined 1.12 Å resolution structure of MCR-2 from I04, provide a starting point for more extensive investigations of the MCR-1 mechanism, which can be used for structure-based design of inhibitors for use in potential colistin-based combination therapies.

Hinchcliffe P et al. doi: 10.1038/srep39392

Insights into a key glycoprotein for vascular health

Endoglin (ENG) is a glycoprotein found in the membranes of cells that line blood vessels in mammals. In humans, it is essential for healthy heart development and blood vessel growth, but also plays a role in preeclampsia and can help tumours establish their blood supply. Although ENG plays a crucial role in the vascular system and its inhibition can slow tumour proliferation, we lack high resolution structural data showing how it is organised and how it interacts with its binding partner, bone morphogenetic protein 9 (BMP9).

Researchers grew and screened several hundred crystals, to gain much-needed structural insights on this protein. Only one crystal yielded a usable dataset, yet the crystal structures it shows offer detailed insights into the mechanism of BMP signalling and inform ongoing efforts to therapeutically target this essential cellular pathway.

Saito T et al. doi: 10.1016/j.celrep.2017.05.011

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The large format goniometer in 103 supporting cornea lens experiments.

Structures and Surfaces

he new Structures and Surfaces Group consists of four beamlines: 105 (Angle Resolved Photoelectron Spectroscopy – ARPES), 107 (Surface and Interface X-ray Diffraction), B07 (Versatile Soft X-ray Scattering – VERSOX) and 109 (Surface and Interface Structural Analysis – SISA). The new structure will also encourage cross group developments and we expect strong interactions with the Magnetic Materials Group, Spectroscopy beamlines and the Soft Condensed Matter Group in a number of science areas such as energy materials or catalysis. The grouping will enhance our ability to work more closely with user groups, offering a comprehensive range of techniques to non-specialists that will aid understanding of their science problems.

The diversity of the science programme is a strength of this group with studies ranging from the observation of novel electronic states in complex crystals to experiments on the structure of lipid layers at the air-liquid interface. This year's science highlights cover two specific areas from this broad range of applications: investigating the mechanism by which ozone reacts with organic molecules in seawater and understanding materials related to spintronics.

Using X-rays to investigate cloud chemistry

Aerosols – tiny particles in the air – are key to the formation of clouds, as all cloud droplets form when water droplets condense around aerosols. If the chemical properties of aerosols are modified, this can affect the size and number of droplets within a cloud, and hence how much light the cloud reflects and how much rain falls. A small chemical change can cause a climatic difference, and although this process has a significant effect on Earth's climate, it is not well understood.

A large number of aerosols are coated with a thin organic film. Previous studies of how these organic films react with atmospheric oxidants, such as ozone, have used proxy organical material. For the first study to use real sample of atmospheric aerosols, researchers employed the unique capabilities of the 107 beamline to capture, via X-ray reflectivity measurements, information on the changes that occurred when the samples reacted with atmospheric oxidants. Their results indicate that aerosols in the atmosphere may be less reactive than previously thought.

Jones SH et al. doi:10.1016/j.atmosenv.2017.04.025

Achieving maximal spin splitting at a crystal surface

The surfaces of materials can host unique electronic properties, where the electrons behave very differently from the interior. All electrons possess a magnetic moment, their spin, but in most non-magnetic materials electrons with different spins behave in the same way. This may not be the case at surfaces, where a splitting of states of opposite spin may be observed. This leads to novel electronic phases, an interesting area of fundamental research but also for potential applications in spin-controlled devices.

For spin-splitting to be useful in electronic devices, it is often necessary to maximise the size of the splitting. Researchers from the University of St Andrews and the Max Planck Institute for Chemical Physics of Solids in Dresden investigated the origin of large spin-splitting on cobalt-based delafossite oxides. They used high-resolution angular resolved photoemission measurements I05, which is ideal for measurements on small samples. Their results show that spin-splitting in delafossite oxides reaches as much as the full strength of the spin-orbit coupling of the relevant orbitals. This highly surprising result is a consequence of unusually strong symmetry breaking at the surface, enable by the crystal structure. This insight could be used to design new materials exhibiting record-breaking spin-splitting.

Sunko V et al. doi:10.1038/nature23898

Understanding the behaviour of electrons in spintronic materials

The interface between magnetic and non-magnetic materials is a key feature of spintronic devices, making it crucial to characterise and understand both the surface and bulk electronic and magnetic properties of the materials used in their production. For transition metal oxides it appears that thicknesses on the scale of several nanometers are critical for the appearance or disappearance of desired properties, but the presence of these critical thicknesses is extremely difficult to assess.

A team of researchers has developed a technique to reliably and precisely detect variations in electronic structure with depth. They used 109, which allows photoelectron spectroscopy at tunable probing depths by changing the photon energy. The researchers applied their technique to two prototypical spintronic materials, and advanced theoretical modelling allowed them to attribute the results to the ability of carriers to move freely though the solid, and to precisely define the depths at which such variations take place. This is a step towards achieving the necessary precise control of spatially-confined spintronic materials.

Pincelli T et al. doi:10.1038/ncomms16051

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Magnetic Materials

he newly formed Magnetic Materials Group concentrates on emergent phenomena in guantum materials using the capabilities of beamlines 106, 110, 116, B16 and 121. Our aim is to realise a family of polarised X-ray beamlines with intuitive software, underpinned by advanced on-site sample characterisation facilities and supported by state-ofthe-art data visualisation tools. Research on our beamlines encompasses a variety of challenges and opportunities at the frontiers of condensed matter physics and materials science ranging from topological states of matter, superconductivity, spintronics, two-dimensional systems, skyrmions and multiferroics. The key insights made by researchers exploit the high sensitivity of polarised X-ray spectroscopy, microscopy and scattering available across the beamlines.

In 2017, studies included investigations into new materials for spintronic devices, and the development of a new pulse picker for synchrotrons.

Imaging exchange coupling in multiferroic, multifunctional devices

The data storage on which our smartphones, tablets and computers rely works via electrical fields (e.g. flash drives) or magnetic fields (e.g. hard drives). Research into magnetoelectric materials could lead to a new generation of multifunctional devices that use a combination of the two.

Magnetoelectric materials have both electrical and magnetic functionality, and changing one induces a change in the other – an effect called 'cross-coupling'. Developing an understanding of cross-coupling involves studying how the magnetic properties change when an electric field is applied. Because most magnetoelectric materials have very complicated structures, Researchers simplified the process by developing a unique process for making a simplier magnetoelectric material.

They then used IO6's X-ray PhotoEmission Electron Microscope (XPEEM) to image – in real time - how changing the electric properties results in a change in the magnetic state. Their results are an important step towards exploiting cross-coupling to make new multifunctional devices that work well at room temperature, opening up new directions for creating low-power 'spintronic' devices that make use of an electron's spin, as well as its charge.

Saenrang W et al. doi:10.1038/s41467-017-01581-6

Adding electrons to switch magnetic chirality

The Dzyloshinskii-Moriya interaction (DMI) was first introduced in 1957 to explain 'weak ferromagnetism'. It causes a twisting of the pattern of atomic magnets, and is now considered to be the driving force behind the exotic magnetic vortexes called Skyrmions. A predictive theory hadn't been developed, however, as until now a detailed study of the amplitude and sign of the DMI hadn't been carried out.

A team of researchers has used 116 to carry out a systematic study of the DMI in a series of magnetic crystals. 116 offers the very high x-ray flux needed to observe the tiny signals associated with magnetism. For these experiments, the researchers installed a movable magnet to drag around the whole magnetic pattern without moving the sample, yielding data of much higher quality than traditional scans.

Their results showed a spectacular change in the magnitude and sign of the DMI as electrons were added to 3d shells, confirming theoretical calculations and a simple model based on the number of electrons carried by the magnetic ion. The study brings us a step closer to systematic prediction of important material properties, and to finding suitable materials for spintronics applications.

Beutier G et al. doi:10.1103/PhysRevLett.119.167201

Fast Active Optics for Synchrotron Radiation

Synchrotron light sources produce a strong and stable pulsed beam, for which the pulse sequence is dependent on the circumference of the storage ring. Many experiments only require some of the pulses, including 'pump and probe' experiments that activate a process and then measure it. Experiment hutches are equipped with pulse pickers that are used to select which pulses reach the sample. Currently, pulse pickers rely on mechanical systems rotating at high velocities, such as crystals or mirrors. They are limited by their rotation speed, and other design factors.

To overcome these limitations, a team of researchers has developed a new type of pulse picker, based on an active optic system that uses surface acoustic waves (SAW) rather than mechanical components. It provides a flexible method of selecting pulses, via electron modulation of the amplitude of the acoustic waves.

For their experiments they used B16, which is equipped with the special optics needed to focus the beam onto the detector. Having successfully demonstrated that this new type of pulse picker is feasible for pulses separated by at least 120ns, the team has plans to install a second one at the BESSY II facility in Germany.

Vadilonga S et al. doi:10.1364/0L.42.001915



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The scientists, engineers, technicians and PDRAs that make up the Magnetic Materials Group.

Imaging and Microscopy

he Imaging and Microscopy Group brings together eight experimental facilities (108, J08, DIAD, 112, 113-1, 113-2, 114 and ePSIC), which use X-rays and electrons to image samples under different experimental conditions across a diverse range of both length and time scales. Different contrast mechanisms allow for imaging of sample properties such as elemental composition, density and structure, and this ability to extract image sample properties in minute detail lends itself to a wide range of scientific areas from chemistry and catalysis to environmental science, materials science, biology, medicine, and cultural heritage.

In 2017 the Electron Physical Sciences Centre (ePSIC) at Diamond welcomed its first users. The collaboration of the expert staff at ePSIC with this range of users is helping to bring cutting edge microscopy techniques to the wider material science community.

Studies this year included investigations into natural carbon sequestration in the Arctic, how fluids move through rocks and the causes of thermal runaway in lithium-ion batteries.

Revealing the reactivity of iron in the Lena River basin

With the melting Arctic ice cap causing its rivers to swell, understanding how organic carbon is transported to the ocean and crucially trapped by these rivers is important for climate change modelling and mitigation. Reactive iron-bearing mineral particles trap organic carbon in soils and continental shelves, yet the amount of reactive iron and how it reacts with organic carbon in large Arctic rivers is poorly understood.

Using a transmission electron microscope and the Scanning Transmission X-ray Microscopy beamline (I08), researchers characterised the size, mineralogy, and form of iron particles from the Lena River and its major tributaries.

X-ray Absorption Near Edge Structure (XANES) spectroscopy and X-ray Fluorescence (XRF) mapping confirmed that iron is transported as particles and colloids in the Lena River, and that 70% of these particles are composed of poorly crystalline ferrihyrite, a reactive form of iron.

The researchers were able to distinguish between carbon and iron at nanoscale resolution, revealing that the ferrihydrite was in the form of discrete particles within networks of organic carbon, and transported by attachment to the surface of larger organic matter (>0.7 μ m) and clay particles.

These important insights show that the Lena River supplies reactive iron to the ocean, a significant proportion of which is already associated with organic carbon. The result confirms iron-bearing particles contribute towards natural carbon sequestration on the continental shelf.

Hirst C et al. doi:10.1016/j.gca.2017.07.012

Investigating lithium ion battery failures

The 18650 cylinder is the most widely used lithium ion (Li-ion) cell geometry, powering everything from laptops to electric vehicles. Its capacity has been steadily increasing, and now more than ever, the safety of Li-ion batteries is of the utmost importance. Cell failure is extremely rare during normal operation, but under abusive conditions the active materials within Li-ion batteries break down exothermically, generating large amounts of heat that can lead to a positive feedback loop, fire and even explosions – a phenomenon known as thermal runaway.

Rupture of 18650 cells during thermal runaway is one of the most hazardous types of failure, and yet the stages leading to rupture are not well understood. Researchers are investigating the cause of rupture to further our understanding of failure mechanisms and to guide the development of safe next-generation Li-ion cells. To capture the dynamic sequence of events with enough detail, they need to use Diamond's high-speed X-ray imaging capabilities. The I12 JEEP beamline offers a large field of view for high speed and high energy imaging of Li-ion battery failures. Their results show that the introduction of a bottom vent on commercial cells can help reduce the risk of rupture and improve the safety of the cell.

Finegan DP et al. doi:10.1002/advs.201700369

Imaging fluid flow in tiny rock pores

Knowing how fluids move through micron-sized voids (or 'pores') in rock kilometres below the Earth's surface is crucial information to help understand and optimise underground carbon dioxide (CO_2) sequestration, oil recovery from reservoir rocks, and a host of other processes. Oil and gas can be trapped in pores as water flows through the rock, with strong trapping beneficial for CO_2 storage and weak trapping preferable for efficient oil recovery. However, the exact nature of trapping at the pore scale is not well-understood, with inhibiting viscous and driving capillary forces both thought to play an important role in controlling flow during trapping events.

To illuminate this process, an innovative flow apparatus was engineered to replicate the high pressures and temperatures seen in reservoirs. Using 113-2, fluid flow was imaged in 3D, at the micron scale, and with a time resolution of around a minute. I13-2 offers photon flux over a million times higher than that of a laboratory X-ray source, perfect for studying rapid dynamic processes like fluid displacement. As a result, the time-resolved imaging provided pore-by-pore analysis of the local capillary pressure during trapping events. Results reveal that water pushes oil into pore spaces in approximately 10 minutes – orders of magnitude slower than the opposite process when oil pushes out water from the pore space. After trapping, the oil rearranges in the pore space to find a new position of minimum energy and the local capillary pressure increases rapidly. These new insights provide useful information to build novel models predicting how these fluids flow and how much oil or gas is trapped.

Singh K et al. doi: 10.1038/s41598-017-05204-4



Studies this year included investigations into natural carbon sequestration in the Arctic, how fluids move through rocks and the causes of thermal runaway in lithium-ion batteries.

Crystallography

he Crystallography Science Group comprises beamlines 111, 115, 115-1 and 119, employing various X-ray scattering and diffraction techniques to study structural properties of crystalline, amorphous, and liquid materials at ambient and different non-ambient conditions. These powerful facilities are used for a wide range of science disciplines, from condensed matter physics, chemistry, engineering, earth and materials to life sciences. Important research fields cover *in situ* studies of batteries, synthesis of new materials as well as structural studies at extreme temperature and/or pressure conditions.

The high demand and productivity of the beamlines are reflected by the output of about 150 publications in 2017. Studies included investigations into the formation of novel materials at high temperatures and pressures, the production of chemicals from renewable resources, and the structure of metal organic frameworks in their glassy state.

Using high temperature and pressure to form novel materials

Chemistry changes at very high pressures, allowing the formation of novel materials. For mixtures of iron (Fe) with a small amount of nitrogen (N), theoretical calculations suggested that new Fe-N compounds would form at high pressures, with enhanced properties such as increased hardness or superconductivity, or the ability to act as a catalyst.

A team of researchers used 115 to investigate these changes experimentally. 115 provides an intense and highly focalised X-ray diffraction beam that allows investigations into the atomic structure of the compounds formed. A new laserheating setup allowed the experiment to reach temperatures of up to 1700°C, and the researchers used diamond anvil cells to achieve pressures of up to 128 GPa, more than a million times atmospheric pressure.

Over the course of their experiments, researchers saw the development of three compounds, ZnS-type FeN, Fe₂N and NiAs-type FeN, which they were able to characterise using X-ray powder diffraction. Above 17.7GPa, NiAs-FeN was the compound formed, and it remained stable as the pressure was reduced, and could be removed for analysis of its physical and mechanical properties. However, they did not observe the iron pernitride FeN₂ that was predicted by the theoretical calculations. Their results will help to increase the accuracy of the theoretical calculations, and will lead to new insights into the intriguing chemistry of Fe-N compounds at high pressures.

Laniel D et al. doi:10.1016/j.jallcom.2017.10.267

Moving towards renewable raw material sources

The world currently relies on coal, oil and gas as raw materials for the production of chemicals. With reserves of these fossil fuels running out, and a growing awareness of the CO₂ pollution their use causes, it is becoming increasingly important to develop sustainable carbon sources. One option is to use biomass (dry plant matter) and a team of researchers have demonstrated a new method for converting biomass into butene gas, which in turn can be processed into the chemicals used in the production of polymers and resins.

Their work uses Gamma-valerolactone (GVL), a chemical processed from biomass raw material, and the catalyst Zn/ZSM-15, and demonstrates that it is possible to use a renewable source material to produce benzene, toluene and xylene.

They used high-resolution X-ray powder diffraction (SRXD) on 111 to examine the structure of their samples, which yielded important information about the reaction mechanism. This was the first time that SXRD had been used to investigate the structures of adsorbed structures of the Gamma-valerolactone GVL and immobilized Zn-species used in the research.

The results are a step towards affordable, sustainable chemical production.

Ye L et al. 10.1002/anie.201704347

Metal Organic Framework Liquids and Glasses

Building on previous work conducted at Diamond, researchers have used X-ray Pair Distribution Function (XPDF) on 115-1 to probe the structure of a Metal Organic Framework (MOF) in its glass state, from which they inferred the structure of its liquid form.

MOFs are crystalline materials. They are receiving a lot of interest due to their porous nature, which allows them to accommodate guest molecules within their structure. Potential applications include drug delivery, catalysis, and the capture or separation of gases. There are around 60,000 MOFs, and until very recently research had focused on their solid form.

For this new study, the team investigated the structure of ZIF-4 at Diamond, the Advanced Photon Source, USA, and ISIS Neutron and Muon Source, UK, using their results to produce a computational model of the glass state from which they could infer the structure of the liquid state.

Their results show that this MOF retains its porous structure in its liquid form. This opens up a whole new area of research, investigating whether other MOFs also behave in this way, as well as discovering the properties and potential applications of liquid and glass MOFs.

Gaillac R et al. doi:10.1038/nmat4998

Studies included investigations into the formation of novel materials at high temperatures and pressures, the production of chemicals from renewable resources, and the structure of metal organic frameworks in their glassy state.



Biological Cryo-Imaging

he recent formation of the Biological Cryo-Imaging Group has brought together dedicated facilities for X-ray, light and electron microscopy at Diamond. The bending magnet B24 is the source of X-rays for the full field cryotransmission X-ray microscope dedicated to biological X-ray imaging and the beamline has also established a cryo super resolution fluorescence microscopy facility, which is a joint venture between Diamond and the University of Oxford. B24 exploits cryo-soft X-ray tomography (cryo-SXT), which is a powerful technique for imaging intact cells in their near native state to resolutions of 25-40 nm. Cryo-SXT provides a unique tool for biologists aiming to understand many key cellular and disease processes, sitting neatly in the resolution gap that exists between electron and light microscopies. Its real power lies in its ability to provide 3D imaging of whole cells with little or no chemical or mechanical modification. B24 is now in the final round of commissioning and optimisation with users and will enter full user operations in autumn of 2018.

The Bioimaging group is completed with the recently established national centre for cryo electron microscopy at Diamond, eBIC (Electron Bio-Imaging Centre). eBIC is the first high-end cryo-electron microscopy (cryo-EM) facility worldwide to be embedded in a synchrotron, and user operations have been set up to mirror the well-established synchrotron beamline model. The centre is funded by the Wellcome Trust, the Medical Research Council (MRC) and the Biotechnology and Biological Sciences Research Council (BBSRC). eBIC has rapidly developed since it welcomed the first user group in July 2015. A key aim of the centre is to provide a state-of-the-art facility for single particle cryo-EM and Ccryo-ET through costeffective, peer-reviewed access based on scientific excellence.

Studies in 2017 included investigations into the mechanism of malaria infection and structure of a photosynthetic complex that harvests and traps infrared light.

How malaria parasites break out of red blood cells

Malaria is widespread in tropical and sub-tropical regions of the world. This highlyinfectious disease is caused by a group of parasitic single-celled microorganisms, which are carried by mosquitoes.

When malaria parasites enter a human via a mosquito bite, they rapidly multiply in red blood cells within a protective casing known as the parasitophorous vacuole. Once the parasites mature they break through this vacuole, as well as the membrane of the red blood cell, to infect other red blood cells. As the exact mechanisms underpinning these crucial stages of infection were unclear, researchers used X-ray tomography at B24 to obtain 3D images of infected cells.

Using special additives to start and stop the breakage of the parasites' double enclosures, they explored the process in detail, and compared it with live cell video microscopy. They also used 3D electron tomography to check the integrity of tiny slices of the infected cells.

The work revealed that the breakthrough process happened abruptly. At a very late stage, the infected host cell underwent a dramatic shape change and then broke, liberating the parasites. Furthermore, the study showed that the membrane of the vacuole became permeable just prior to its breakage. These new insights could help to develop novel anti-malarial therapies that target this critical process.

Hale VL et al. doi: 10.1073/pnas.1619441114

Red light photosynthesis

In photosynthesis, light harvesting (LH) complexes funnel absorbed solar energy to reaction centre (RC) complexes, which transiently trap and store the harvested energy in the form of a charge separated state. Thereafter, a series of electron and proton transfers within the RC converts a guinone acceptor to its reduced form, a auinol.

Plants and algae usually absorb light energy at wavelengths of up to 700 nm, but one particular bacterium can absorb and use wavelengths far greater than these. Remarkably, these bacteria have adapted RCLH complexes with the capacity to absorb energy above 1000 nm: near infrared light that is inaccessible to other photosynthesisers.

A team of researchers used cryo-EM at eBIC to determine the structure of the adapted RCLH complex. Their results showed that the bacterium employs an intriguing range of strategies to allow it to harvest light at 1000 nm. The structural insights gained from this study could enable near infrared light absorption to be engineered into biosynthetic, bioinspired or biohybrid photosynthetic structures. In particular, bacteria could be adapted to have a wide input of solar energy to be used for solarpowered cell factories.

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The team will continue looking at this bacterium, and they plan to build on the huge success of this structural study and explore other photosynthetic systems using cryo-EM.

Qian P et al. doi: 10.1038/s41586-018-0014-5



DIAMOND LIGHT SOURCE ANNUAL REVIEW 2017/18

harvests and traps infrared light.

Spectroscopy

he Diamond Spectroscopy Group is built around four beamlines; the Microfocus Spectroscopy beamline (118) the Core X-ray Absorption Spectroscopy (XAS) beamline (B18) and the two independently operating branches of the Versatile X-ray Absorption Spectroscopy beamline, 120-Scanning and 120-EDE. Each beamline has its unique characteristics and they are optimised for performing different types of experimental studies. Over the last year more than 1,350 peerreviewed experimental shifts were delivered using the Spectroscopy Group beamlines, covering many different scientific disciplines, from chemistry and catalysis to environmental and life sciences, materials science, hard condensed matter and cultural heritage. Studies this year include two investigations into industrially-important catalysts and research into a key protein of metabolism regulation.

Atomically dispersed gold species can act as catalysts

Mercury is one of the most toxic substances on the planet, and the UN recently ratified the Minamata Convention, an international treaty designed to protect human health and the environment from anthropogenic emissions of mercury and mercury compounds. The current production process for vinyl chloride, the monomer used in PVC and everything from piping and tubing to gels and lubricants, relies on a mercury-based catalyst, and a more environmentally-friendly alternative is now required.

Gold supported on carbon is currently being commercialised as an alternative catalyst for this reaction. However, its behaviour under harsh reaction conditions has never been investigated, and the reaction is typically carried out at 200°C, using corrosive gas mixtures that can rapidly deactivate catalysts.

Researchers used X-ray Absorption Spectroscopy (XAS) on B18 to observe the reaction *in situ*, and their results offer the first example of a single site catalyst remaining atomically dispersed and working under such harsh reaction conditions. This work will enable more rational catalyst design to find a replacement for the current mercury catalyst.

Malta G et al. doi: 10.1126/science.aal3439

Probing catalytic structure

The Fischer-Tropsch process, used to convert hydrogen and carbon monoxide to hydrocarbons, relies on the use of catalysts. These make the process more efficient, and can control which products are produced, from long hydrocarbon chains to heavy waxes.

Researchers have developed a novel technique to investigate how cobalt catalysts change during the Fischer-Tropsch process. Using the Microfocus Spectroscopy beamline (118) allowed them to focus X-rays down to the microscopic sizes (5 µm) needed for the study, and they performed the first simultaneous measurement of all the characterisation techniques (absorption, diffraction, fluorescence, mass spectrometry) on the beamline.

Investigating the influence of the deposition sequence on the chemical and physical structure of the catalyst, in 3D, under conditions close to those used in large scale reactors, ensured that the observed structure and activity relationships from the experiment are relevant in the real world. The results showed that the performance of the catalysts can be significantly affected by even small modifications in how they are made.

Price SWT et al. doi:10.1126/sciadv.1602838

Understanding the structure of cytochrome c

The cytochrome c protein is essential for human metabolism. It plays a role in the regulation of mitochondrial activity, which allows cells to adapt to changing conditions and to control oxidative stress. Some diseases, including cancer and ischemia (an inadequate blood supply to an organ or part of the body) involve reversible phosphorylation at a particular site on the cytochrome c protein. An international team of researchers has used a variety of experimental methods to analyse the structure, dynamics and functional features of an engineered, stable variant of cytochrome c, in order to understand the molecular basis underlying the effects of this phosphorylation.

The researchers chose I20 to carry out X-ray Absorption Spectroscopy (XAS), because the I20-Scanning branchline is able to analyse highly diluted biological samples. They used XAS to determine the protein's structural features. Nuclear Magnetic Resonance (NMR) showed that only local structure changes were induced by cytochrome c phosphorylation, with the overall protein conformation hardly affected. XAS and NMR both showed an increase in the mobility of the local protein region, which explained the functional changes observed. These findings provide a framework for further investigation of the modulation of mitochondrial activity by phosphorylated cytochrome c, and for the development of novel therapeutic approaches.

Moreno-Beltrán B et al. doi:10.1073/pnas.1618008114

Over the last year more than 1,350 peer-reviewed experimental shifts were delivered using the Spectroscopy Group beamlines, covering many different scientific disciplines, from chemistry and catalysis to environmental and life sciences, materials science, hard condensed matter and cultural heritage.



	0.5904
	0.5166
	0.4428
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Soft Condensed Matter

he Soft Condensed Matter Group provides the infrared (IR) and circular dichroism (CD) spectroscopy and X-ray scattering imaging capabilities, via four beamlines B21, B22, I22 and B23. This unique portfolio of beamlines can analyse a range of samples that include two-dimensional thin films (photovoltaics), living mammalian cells, three-dimensional matrices (metal-organic frameworks) and nano-particles in non-crystalline states.

The Soft Condensed Matter Group maintains a dedicated laboratory space for visiting users, with equipment for sample preparation and analysis such as a centrifuges, a small tissue-culture facility, spectroscopy equipment and the ability to work with different gases. Since the start of 2017, both B21 and B23 now offer mail-in services for solution-state Small Angle X-ray Scattering (SAXS) and CD measurements.

Studies this year included ground-breaking work towards tackling a common, and often fatal, form of cancer, controlling protein crystals with electrochemistry, and building novel materials with DNA.

Disabling leukaemia's defence mechanism

Acute myeloid leukaemia (AML) is one of the most common cancers of children and the elderly, originating from white blood cells in the bone marrow. Current treatment options consist of aggressive chemotherapy and stem cell transplantation, however the disease is often fatal.

Scientists hope that by investigating the molecular mechanisms employed by AML cells to proliferate and avoid detection by the immune system will lead to better treatments. A team of researchers used Synchrotron Radiation Circular Dichroism (SRCD) spectroscopy at beamline B23 to probe the interactions of proteins involved in a molecular pathway responsible for AML escaping an immune response. They found that this pathway is unique to AML and does not exist in healthy cells.

Targeting this pathway could significantly enhance patients' immune defences, offering the hope of a fundamentally new strategy for treating AML.

Gonçalves Silva I et al. doi:10.1016/j.ebiom.2017.07.018

Shocking protein crystals into action

Revealing protein structures is essential to understanding how they work. X-ray crystallography is a valuable tool for providing 'snapshot' images of working proteins at an atomic level, but it can be tricky to 'freeze' some proteins at particular moments in time.

NiFe hydrogenase enzymes are proteins that allow microbes to survive using hydrogen gas as their only energy source. They have potential applications in green energy technologies, but to fully determine how they work we need to image them at specific stages of their reactions.

NiFe hydrogenases contain markers that are easily studied using infrared light, which makes them ideal subjects for the Infrared Microspectroscopy beamline (B22). Crystal forms of the enzymes were analysed within a custom-built electrochemical cell that slowed down their reactions, allowing researchers to study them in unprecedented detail.

The demonstration that hydrogenase protein crystals could be controlled using electrochemistry is an important finding as many other proteins can be studied in the same way, including proteins that convert carbon dioxide into useful chemicals.

Ash PA et al. doi:10.1039/C7CC02591E

Building 3D nanomaterials with sticky DNA bricks

Novel materials with a finely organised 3D nanoscale structure can be used in everything from energy storage to biomedicine. These materials have to be assembled from the bottom up, but as the building blocks involved are too small to handle, the materials must be encouraged to self-assemble.

Nanomaterials based on DNA have been limited in size and shape. To overcome this barrier, researchers have developed a new type of DNA nanomaterial, named C-stars. It is made from single-stranded DNA molecules adorned with cholesterol molecules to help them stick together into crystals.

Analysis of the crystals at the Small Angle Scattering and Diffraction beamline (122) showed that the C-Stars adopt a crystal lattice arrangement. This lattice geometry can be changed by altering the shape of the C-Stars, and the DNA network can be engineered to change its structure in response to an external stimulus.

With such a flexible and robust building material, the resulting crystal arrangements are almost limitless. The 3D crystals are a completely new class of DNA nanostructures, which will have applications in advanced technologies such as biosensors, light harvesting and molecular sieving.

Brady RA et al. doi:10.1021/acs.nanolett.7b00980

Studies this year included groundbreaking work towards tackling a common, and often fatal, form of cancer, controlling protein crystals with electrochemistry, and building novel materials with DNA.



Industrial Liaison

The past year has been another year of growth for the industrial user programme at Diamond. From across the globe 50 companies are making regular use of the beamlines, and increasingly the cryo-electron microscopy (cryo-EM) facilities. The Industrial Liaison team are supporting around 250 different experimental sessions with industrial users each year, with the majority of users coming from the life sciences sector.

The growth in industrial usage by life science companies has been supported by the expansion of Diamond's capabilities in this area and we are now able to offer a wider range of structural biology services than ever before. Recent developments in detectors and the latest generation of microscopes has meant that previously unattainable resolutions are now routinely possible through cryo-EM at Diamond's Electron Bio-Imaging Centre (eBIC).

In an effort to reduce barriers to innovation, the Industrial Liaison team have been working with colleagues across STFC in two funding schemes which aim to help companies overcome intractable product, manufacturing or process performance problems through advanced measurement and analytical technologies.

The first scheme was funded through Innovate UK and STFC plus other external partners such as the National Physical Laboratory. Analysis for Innovators (A4I) opened to applications in January 2017, allowing companies to apply for a share of £6.5 million to work with facilities such as Diamond on solutions for analysis and measurement problems. Two projects were funded to use Diamond, including one with a micro-SME, Lewtas Science & Technologies, studying "phase change control by very low power ultrasound".

We are enabling industrial access to the full wealth of technology and knowledge available at Diamond by extending our services to the technical expertise of our Optics and Metrology Group in addition to our beamlines and microscopes. To date, commercial suppliers of synchrotron optical systems have been able to benefit from Diamond's state-of-the-art metrology instruments and facilities that facilitate testing to unprecedented levels.

As well as growing the industrial programme here at Diamond, the Industrial Liaison team has been active in a number of international collaborations. Within the EU, the team participates in CALIPSOplus, a project funded through the European Union's Horizon 2020 Research and Innovation programme. Further afield, the team have been awarded funding via the Newton Fund for projects in Thailand and Indonesia. The Newton Fund's aim is to develop science and innovation partnerships that promote the economic development and welfare of collaborating countries.

If you would like to learn more about any of these recent developments, please do contact us on industry@diamond.ac.uk.





Collaborations Update

ollaborations support and enhance the work of the Diamond synchrotron, and build Diamond's expertise and reputation as a provider of world class science.

The UK Catalysis Hub

Catalysis is a core area of contemporary science, engineering and technology, with substantial economic and societal impact. Rooted in chemistry and chemical engineering, catalytic science is now multidisciplinary, drawing strongly from materials and bio-sciences.

The Hub is a national network with over 35 collaborating universities, with a physical base at the Research Complex at Harwell (RCaH). Established in 2013 with funding from EPSRC, its aim is to coordinate, promote and advance the UK catalysis research portfolio. The project has five themes each directed by a lead investigator from five partner universities:

- Catalyst Design: Led by Prof Richard Catlow, UCL and based in the RCaH
- Catalysis for Energy: Led by Prof Christopher Hardacre, Queen's University Belfast
- Catalysis for Chemical Transformations: Led by Prof Matthew Davidson, Bath
- Environmental Catalysis: Led by Prof Graham Hutchings, Cardiff
- Bio Catalysis and BioTransformations: Led by Prof Nick Turner, Manchester

The Hub provides a platform for researchers to work collectively and gain frequent access to the Diamond synchrotron, and other facilities at Harwell. A whole system approach to the study of catalysis, combined with high throughput, allows optimal experiments to be carried out that shorten the path to development of commercially useful products, and promote the UK catalysis effort and expertise on a global stage. The Hub has strong links with industry, which are coordinated by an Industrial Advisory Panel comprising several UK and international industrial members.

The UK catalysis Hub also presents a training experience, with a number of PhD students placed at the RCaH working on catalysis projects with the Hub. This initiative forms part of the Hub's aim to develop the next generation of catalytic scientists, through courses, conferences, PhD programmes, summer schools and outreach activities.

Diamond Manchester Collaboration

The collaboration between the University of Manchester and Diamond Light Source was established to construct and operate an imaging facility at Diamond's X-ray Imaging and Coherence beamline (I13). Complementary to the Coherence branchline (I13-1), the Diamond Manchester Imaging branchline (I13-2) performs real space imaging and tomography on a length or timescale not achievable in home laboratories. Thanks to the high flux from Diamond, images of high signal-to-noise ratios can be recorded very quickly on 113-2. This enables the study of dynamic processes such as electrochemical deposition for explaining battery failure, bubble dynamics in molten metals for understanding how metals solidify and therefore fail, and the dynamics of the closed cochlea, the part of the ear that converts sound waves to electrical signals.

Manchester financially assisted the building of the 113-2 branchline and continues to contribute to its staff, operations and development. Diamond owns and has overall responsibility for the branchline, and provides all other funding. In return for its investment, Manchester has guaranteed access to beamtime at Diamond. The majority of this is carried out on 113-2, but substantial amounts of beamtime are available on other beamlines. Manchester uses 113-2 for research that spans materials science, biomedicine, geology and engineering, as well as methods to develop the fields of X-ray imaging and tomography. To complement the work undertaken at Diamond, Manchester has set up a chapter of the Manchester X-ray Imaging Facility at the RCaH. To date the collaboration has produced 78 publications.

The partnership formally began in May 2010 and will continue until at least 2020. Both partners view the collaboration as a long-term, ongoing relationship, working together as key national players in X-ray imaging and tomography. The Diamond Manchester collaboration has been involved in many other partnerships including US National Labs, Advanced Light Source, ESRF, CLF and the nuclear industry.

Education, Training and Engagement

iamond continued its extensive engagement programme throughout 2017-18, linking world-leading science and engineering professionals with a diverse range of audiences. Our events welcomed 5,635 visitors to the facility: 1,413 for scientific and technical events, 668 undergraduate and postgraduate visitors, and 3,554 school students and members of the general public.

The majority of our visitors come for Diamond's core program of events, including our popular Inside Diamond open days, which give members of the public a chance to visit the facility in small groups, to see inside the synchrotron and visit a scientist at their beamline.

"From start to finish the atmosphere was great with many people on hand to chat ... The tour around the facilities was also most informative with some aspects and numbers producing jaw dropping reactions from members of the group."

Diamond open day visitor



Regular Schools open days give A-level students a general overview of the facility and highlight career opportunities available to them. Dedicated activities allow students to experience specialist areas within the facility including biology, computing, and engineering. The Diamond schools work experience program took its third cohort in 2017, offering 28 students an immersive week of project-based work.

In September we partnered with Harwell Campus and the Medical Research Council to host a stand at the New Scientist Exhibition at the ExCel Centre in London. Over 30,000 people attended the four day event, many working in areas of science and engineering. The Bluedot music festival at Jodrell Bank Observatory, where music is combined with a programme of live science and expert talks, provided the opportunity to engage with a very different audience. Thousands of festival goers were able to visit our exhibition stand and talk to some of Diamond's summer placement students about their projects and wider Diamond activities.

Engaging with Higher Education

Our work placement schemes for undergraduates continue to provide students with in-depth research projects, whilst also developing life-long skills in communication, engagement and project management. In June 2017, six Year in Industry and 14 summer placement students arrived at Diamond to begin their projects. Whilst based at Diamond, the students also had opportunities to visit other research teams and facilities on-site and to present their research to the wider Diamond organisation.

Diamond welcomed 25 new PhD students in 2017, bringing the total number of co-funded PhD studentships to over 90. We proudly collaborate with research organisations around the UK and beyond, hosting co-funded PhD students and training the synchrotron scientists of the future. Continuing from the successful introduction of a centralised call for collaborative PhD proposals in 2016, the 2017 scheme received 59 proposals. Following an internal review process, 16 studentships were funded and we look forward to welcoming our new students starting in October 2018.

The demand for visits from the university student community continues to increase and Diamond met this by hosting 26 postgraduate and undergraduate groups from across the country in 2017.

Scientific Workshops and Conferences

In 2017 Diamond hosted a range of events targeted to meet the interests and development needs of our staff and user community, including workshops, conferences and training schools relating to specialist fields, techniques and software.

Diamond hosted three international conferences, each from an established conference series: the 20th International Magnetic Measurement Workshop (IMMW20), a satellite workshop of the IUCr Congress meeting (Q2XAFS) and the ninth International Workshop on Infrared Microscopy and Spectroscopy with Accelerator Based Sources (WIRMS 2017).



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