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Status of the crystallography beamlines at Diamond Light Source*

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Abstract. X-ray crystallography is one of the most widely supported areas of science at Diamond Light Source and is the core technique on a third of the beamlines, accounting for almost a half of user publications across the whole facility. The life science community can currently call on six macromolecular crystallography beamlines (I02, I03, I04, I04-1, I23 and I24) to satisfy the increasing demand for the determination of macromolecular crystal structures from both the academic and pharmaceutical communities. In the physical sciences, there are four beamlines (I11, I15, I16 and I19) that cater for crystallographic studies ranging from the structure solution of small-molecule systems, the determination of structure at extreme conditions and the detailed analysis of the weak diffraction signal from magnetic structures. The physical science beamlines use both single-crystal and powder-diffraction techniques. All the crystallography beamlines at Diamond ensure they undergo a continuous programme of development to enable new technologies and the science they support. In this article, the current status of all the crystallography beamlines at Diamond will be discussed along with some examples of the science they have supported since they became operational.

1 Introduction

Diamond Light Source is a medium-energy third-generation synchrotron based in Oxfordshire, UK. The organisation operating the facility, Diamond Light Source Ltd (DLS), was constituted with the signing of a Joint Venture Agreement, between the Wellcome Trust and the CCLRC (on behalf of the UK government), on 27th March 2002. DLS is headed by its Chief Executive Prof Andrew Harrison and its Board of Directors under the chairmanship of Sir Adrian Smith. The UK's Science and Technology Facilities Council (STFC) own 86% of the shares in DLS, with 14% of the shares being owned by The Wellcome Trust. The initial phase of construction of Diamond, Phase I, was completed in 2007, with 7 beamlines operating for the user community. The storage ring consists of 24 double-bend achromat cells and has a circumference of 561.6 m. It operates at an energy of 3 GeV with a beam current of 300 mA. The 158.4 m circumference booster synchrotron receives electrons at 100 MeV from the linac and injects these at the full 3 GeV operating energy into the storage ring. The booster refreshes the electrons in the storage ring via top-up mode at 10 minute intervals to provide a quasi-continuous beam current. At the time of initial operation, the storage ring had the lowest emittance, at 2.7 nm-rad in the horizontal and 0.03 nm-rad in the vertical, of any 3rd-generation source. Phase II of construction (2007-2013) provided 15 further beamlines and Phase III will run until 2018, ultimately delivering a total of 33 beamlines. Throughout the period of operations the beam current and reliability have ramped up, while delivering more experimental shifts and attracting and supporting increasing numbers of users. This includes a very significant component of industrial users, and from the start of user operations Diamond established an advisory body for industrial access and development —Diamond Industrial Science Committee (DISCo)— which draws on senior figures or key users from across all relevant industrial sectors, and has proved very effective in advising Diamond how best to develop its facilities for the needs of industry. Over 70 companies have now made use of Diamond through the proprietary access mode, with additional companies coming to Diamond through peer review.

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Diamond offers a range of access options for academic users. *Direct access* mode provides regular access to individuals or groups with two proposal calls issued per year. The deadlines of the call for direct access proposals are April 1st (for the period of 1st October, of the same year, to march 31st, of the following year) and October 1st (for the period of 1st April to 30th September of the following year). So that long-term access to a team or consortium can be provided, *programme mode access* is also available. There is one call for proposals during the year with a deadline of 1st April, which awarded beamtime covering the period from October (of the same year) for a period of 2 years. Finally, *rapid access* mode is available on some beamlines for urgent projects which require more immediate use of the facility.

Diamond currently hosts twenty six operational beamlines with a further four under construction. At the time of writing, there is a suite of five operational beamlines for macromolecular crystallography (MX), one nearing completion and a further two beamlines in the design stage. The MX beamlines I02, I03 and I04 were in the phase-I tranche of beamlines built at Diamond and were among the earliest to be made available for user operations. As will be described in more detail later, these first three MX beamlines are virtually identical in design and offered users at the time state-of-the-art facilities for high-throughput protein crystallography. They have all subsequently undergone substantial upgrades which have dramatically improved their throughput. Beamline I24, a beamline offering MX users microfocussed X-rays, and beamline I04-1, a fixed-wavelength side-station to I04, were built in phase-II of Diamond's beamline construction. At the time of writing, I24 is undergoing a substantial upgrade to its end-station. The most recent beamline to be constructed, in the final phase-III build, is the long-wavelength MX beamline I23 which offers users the opportunity to collect data at X-ray energies suitable to detect the anomalous signals required for phasing from the sulphur or phosphorous atoms that are present in native protein or RNA/DNA crystals.

In the Physical Sciences, Diamond currently has four beamlines that are used for X-ray crystallography. The extreme conditions beamline, I15, was built in the phase-I construction period and, unlike the other crystallography beamlines which use undulator sources, it has a wiggler that produces the very high-energy X-rays required to penetrate pressure cells and other sample environment equipment. As we shall see it can be operated in both powder-diffraction and single-crystal diffraction modes. The dedicated powder-diffraction beamline, I11, was built in phase-II and has just been upgraded to include an additional experiments hutch, which will offer a facility for long-duration experiments that require very short periods of beamtime over regular periods spanning weeks to months. Beamline I16, the Materials and Magnetism beamline, was built in phase-I and provides a single-crystal diffraction facility for the study of magnetic structure and detailed diffraction phenomena, including coherence effects, around individual Bragg reflections. Finally, I19 is a beamline dedicated to small-molecule single-crystal diffraction techniques. As well as supporting core chemical crystallography research, the beamline also provides facilities for sample-environment studies, including instrumentation for high-pressure experiments and the study of gas exchange in porous materials, as well as facilities for time-resolved experiments. Beamline I19 was built in phase-II and, after six years of user operation, it will receive a major upgrade in early 2015.

2 The life sciences beamlines

2.1 Beamlines I02, I03, I04 and I04-1, high-throughput and highly automated beamlines for macromolecular crystallography

The high-throughput protein crystallography beamlines I02, I03 and I04 were all built at the same time, during phase-I of beamline construction at Diamond, and all three have essentially the same design [1,2]. They all use 2 m *in-vacuum* undulators with I02 and I04 having a 23 mm period while I03 has a period of 21 mm and is the only beamline at Diamond that offers facilities for the handling of biological agents in Hazard Groups 3. The layouts of the optics hutches are all extremely similar and they are equipped with cryocooled double-crystal monochromators, manufactured by FMB Oxford, and bimorph horizontal and vertical focusing mirrors, which were fabricated by Seso and installed onto the mechanics and vacuum vessel supplied by FMB Oxford. The mirrors each have three stripes, bare fused silica (or Si), and sections coated with Pt and Rh, to provide optimised reflectivity and harmonic rejection across the available energy range. For the 2.73 mrad grazing incidence angle of the mirrors, each stripe provides essentially harmonic-free X-ray beams in a specific energy window: *i.e.* 5–10 keV for the silica stripe; 10–22 keV for the rhodium stripe; and 22–25 keV for the platinum stripe. The focusing performance of the mirrors varies from beamline to beamline but, on average, the focused beam profile at the sample position has horizontal and vertical dimensions of approximately $70\,\mu\text{m} \times 20\,\mu\text{m}$ (FWHM), respectively. Compound refractive lenses (CRLs) are available on all three phase-I beamlines. These have been used on I02 and I03 in conjunction with the bimorph mirrors to affect a focus of $35 \times 10\,\mu\text{m}$ beamsize at discrete energies. On I04 these have been used without mirrors to provide energy dependent beamsizes that vary from $8 \times 3\,\mu\text{m}$ to $200 \times 200\,\mu\text{m}$, bringing microfocussed beam to the phase-I beamlines. All three beamlines are also equipped with fixed apertures just prior to the sample position and allow beam shaping of 50, 20 or $10\,\mu\text{m}$ depending on the specific beamline. The beamlines are equipped with Rigaku Actor sample changers capable

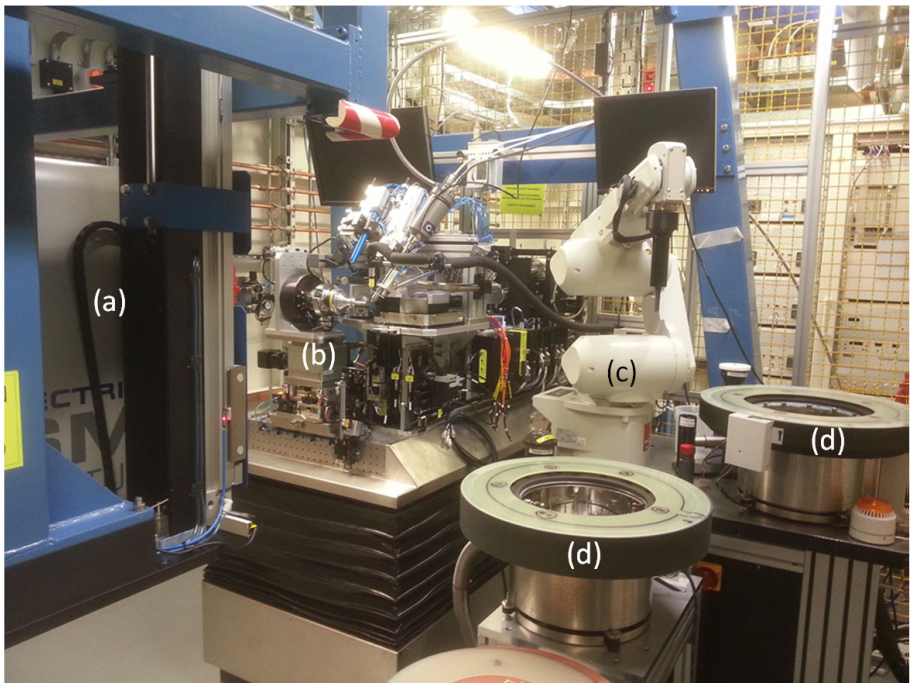

Fig. 1. The end-station for macromolecular crystallography beamline I04: (a) Pilatus 6M detector; (b) goniometer stage; (c) Rigaku robotic sample changer; (d) sample dewars.

of holding 160 samples on SPINE [3] standard mounts in 10 Unipuck containers. Sample exchange time is in the order of 30 seconds. All Diamond MX beamlines use the Unipuck system with SPINE standard pins.

The experiments hutches all have a virtually identical layout. The detectors are mounted on a motorised platform, which is itself supported by a large A-frame-type structure. The assembly provides $\sim 10\,\mu$m positional stability for detectors that could weigh up to as much as 100 kg. Originally the beamlines had ADSC Quantum 315r CCD detectors but these have all now been replaced by Pilatus 6M-F or Pilatus 3 6M photon counting detectors, allowing shutterless operation. After approximately four years of operation, preceding the detector upgrades, the end-stations of I02, I03 and I04, including the goniometry, sample viewing optics and beam conditioning elements, were all substantially upgraded to improve functionality and, as discussed, the inclusion of microfocusing capabilities via compound refractive lenses, fig. 1. Currently the single-axis goniometer on I04 hosts a mini-kappa. The goniometers have spheres of confusion that are in the range of 1–2 μm and the overall sphere of confusion on I04 with the mini-kappa mounted is 4–6 μm depending on the kappa setting. Note that the crystal needs to be recentred at each new orientation of the phi and kappa axes. As part of the phase-II beamline construction, the optics hutch of I04 was adapted to accommodate the front end and monochromator for the I04-1 side station.

I04-1 has a fixed energy (13.53 keV which is equivalent to ~ 0.916 Å) and although its experiments hutch is located on the floor space between I04 and I03 (in the position that would otherwise have been occupied by B03) it has its own U28 ex-vacuum undulator source. This is positioned just upstream of the I04 undulator, in the same straight section of the synchrotron, and a vertically mounted monochromator crystal housed within the I04 optics hutch deflects the beam horizontally into the I04-1 experiments hutch. The monochromator was manufactured by Instrument Design Technology (IDT). The beam is focused by a vertically deflecting toroidal mirror supplied by Cinel and Winlight. The I04-1 experimental instrumentation is aligned at the same offset angle within the experiments hutch, almost corner to corner, while the hutch itself is neatly positioned within its fan between the other beamlines. The side station, despite offering only a fixed wavelength, has the benefits of the high flux and low beam divergence of an undulator and has been built to help address the over subscription of MX beamtime. The end-station is based around an Accel MD2 goniometer, equipped with a mini-kappa, and the beamline currently has a Pilatus 2M detector, for shutterless data collections, though this will shortly be upgraded to the larger Pilatus 6M-F. The end-station has an Irelec CATS robotic sample changer [4], which handles the same pucks as the other MX robots. The goniometer has a 2 μm sphere of confusion in single-axis mode and a 4 μm sphere of confusion when the mini-kappa device is in use (once the crystal has been re-centred at each new orientation of the phi and kappa axes). The detector is mounted on a motorised tower, which can vary the sample to detector distance, instead of an A-frame, fig. 2.

Perhaps the most radical change to have occurred to the original suite of MX beamlines at Diamond has been the recent reconfiguration of beamline I02 to accommodate the Versatile MX beamlines VMXi and VMXm which will be built in late phase-III. The machine lattice at this point, at the cell incorporating the I02 front-end, will be

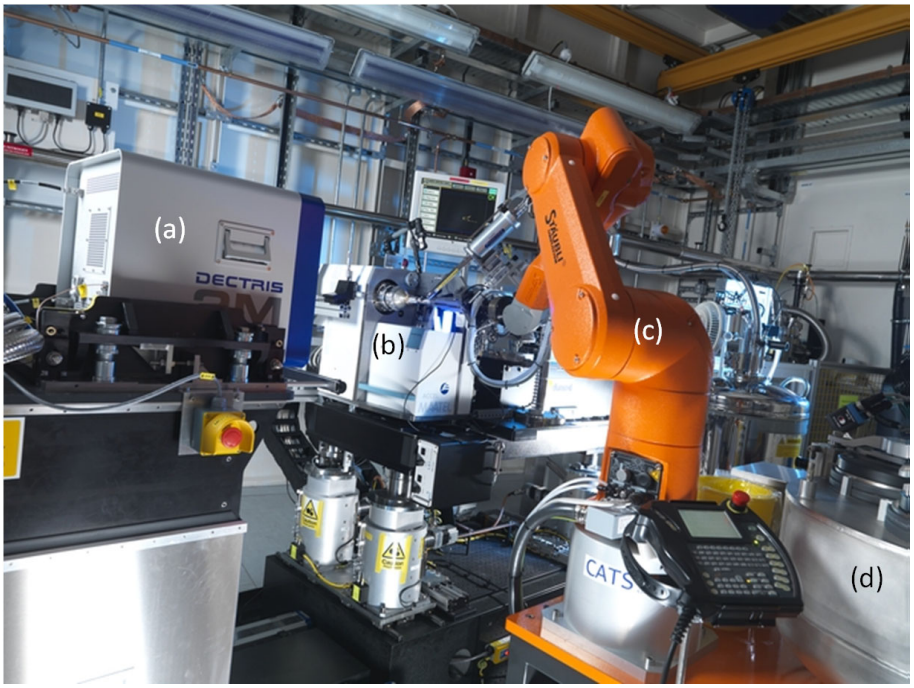

Fig. 2. The end-station for the macromolecular crystallography beamline I04-1: (a) Pilatus 2M detector; (b) Accel MD2 goniometer; (c) CATS robotic sample changer; (d) sample dewar.

reconfigured into a double-double bend achromat (DDBA) arrangement, maintaining the insertion device position of the current I02 beamline which will be reused in VMXi (*in situ*) and making it possible to add another insertion device at the current bending magnet position BM02 which will be used for VMXm (microfocus). VMXm aims to provide beamsizes in the range 0.5–5.0 μm pushing the boundaries of micro crystals at Diamond. VMXi will become a beamline permanently dedicated to collecting data from *in situ* in crystallisation containers. This new beamline is still very much in its conceptual design stage but it will reuse the optics hutch of I02 and its experiment hutch will be directly downstream of that of I02. Currently, I02 has a temporary control room placed adjacent to its modified experiments hutch.

An example of the work carried out on the MX beamlines is amply illustrated by a study optimising drug targets. Micco and co-workers [5] used beamline I04 in a drug design study where they evaluated binding of tetra-substituted naphthalene diimide (ND) ligands to G-quadruplexes, which are guanine-rich four-stranded DNA secondary structures. In a structure-based design approach using information from previous studies, the group could find new high-affinity ligands and were able to co-crystallise these with telomeric quadruplex DNA sequences and solve the structures of three ligand-DNA complex compounds. ND ligands are of interest since they show a high potency, both *in vitro* and *in vivo*, against cancer cell lines, *e.g.* pancreatic MIA PaCa-2 and are thus potential drug targets in cancer therapy. The crystallographic studies helped to optimise the chemical structure of the ligands which led to an enhancement of their pharmacological properties. The binding mode of one of the ligands to the DNA sequence is shown in fig. 3.

2.2 Beamline I24, a high-throughput variable microfocus beamline for macromolecular crystallography on crystals of only a few microns in size

Beamline I24, which became operational in mid phase-II, has been designed with microfocusing optics to provide beam sizes ranging from 5 μm to 50 μm so that full structure determinations can be obtained from very small crystals [6]. Along with I03 this beamline can handle containment level 2 samples. For a reasonable chance of a successful outcome on the other MX beamlines, crystals with dimensions greater than $\sim$ 50 μm are considered ideal but there is an increasing number of cases where crystals of this size are difficult, if not impossible, to obtain. Membrane proteins, for example, are notoriously difficult to crystallise and easily fall into this "awkward customer" category. Although they are essential for a very large variety of biological functions, including molecular transport, the relay of signals and respiration, and they compose up to 30% of the proteins encoded by eukaryotic (nucleus containing) cells, there are less than 20 independent structures of integral membrane proteins currently reported. To put this into perspective the number of structure determinations of all proteins now runs into the many thousands. In part, I24 was built to meet the very demanding challenges of determining structures from very small membrane protein crystals.

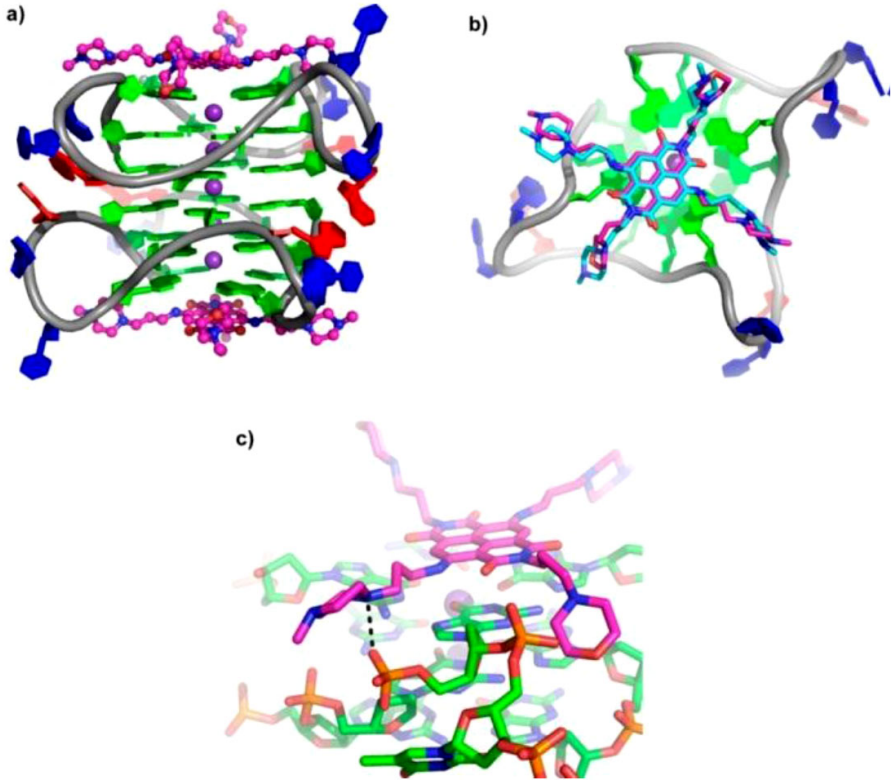

Fig. 3. (a) Cartoon representation of complex A. Potassium ions are shown as small spheres. Two asymmetric units are shown with two 5′–5′ stacked intramolecular G-quadruplexes with compound 3d (purple) bound to external 3′ G-quartet surfaces (PDB 3UYH, 1.95 Å resolution). (b) Structural alignment of 3UYH and 3SC8 (both with 447 atoms) (rmsd = 0.76 Å). Compound 3d is shown with carbon atoms colored mauve, and compound 4 has carbon atoms colored cyan. (c) View of complex A with compound 3d, showing possible electrostatic/hydrogen bond interactions between a phosphate group and the terminal nitrogen atom of an N-methyl-piperazine group.

Beamline I24 uses an *in-vacuum* U21 undulator as its source and its general layout, including double-crystal monochromator and horizontal and vertical bimorph mirrors is broadly similar to those of I02, I03 and I04. However, the first set of mirrors is primarily for pre-focussing the beam onto an aperture, or virtual source, rather than the sample. This virtual source is then imaged by a set of micro focusing mirrors placed further downstream and this second set of mirrors can be translated slightly along the beam direction to allow the beam size to be varied rapidly. For I24, the monochromator was supplied by Bruker (formerly Accel) while Seso produced both the bimorph pre-focusing and micro-focusing mirrors, which are mounted within the mechanical elements manufactured by FMB Oxford. To optimise the performance of the optics, the single-axis goniometer is positioned as close to the exit port of the vacuum vessel of the microfocus mirrors as possible and the remaining beam conditioning components, and the sample viewing camera, occupy the extremely compact space left between them. The end station is otherwise broadly reminiscent of the other MX beamlines. Originally the beamline was equipped with a Rayonix MarMosaic300 detector which was replaced with a Pilatus 6M-F, to allow shutterless operation, which in turn was replaced by a Pilatus 3 6M. As on beamline I04-1, an Irelec CATS sample changer is used for fully automated sample exchange [4], see fig. 4. Currently this allows exchange of cryo-cooled samples in less than 45 s.

Currently, I24 has been temporarily withdrawn from user operations as it is undergoing a complete replacement and upgrade of its end station. The upgrade will introduce an in-house designed vertically oriented omega rotation stage for conventional crystallographic studies, with crystals mounted individually to sample loops, with sub-micron positional and rotational accuracy. A second horizontally oriented stage will be used for studies of samples contained within crystallisation trays —so-called *in situ* studies.

Of the many scientific highlights to come from I24 is the work that the beamline supports on the determination of the structure of viruses. For example, crystal structures determined at I24 have helped to design a new methodology for production of a vaccine for foot-and-mouth disease virus (FMDV). X-ray crystallography carried out using the *in situ* setup showed that stabilised and wild-type empty capsids have essentially the same structure as the intact virus. Elucidation of the structures both the wild type and H2093C form of the virus confirmed that point mutation of a histidine (highlighted in fig. 5) to a cysteine residue in the VP3 protein results in the formation of a disulphide bond.

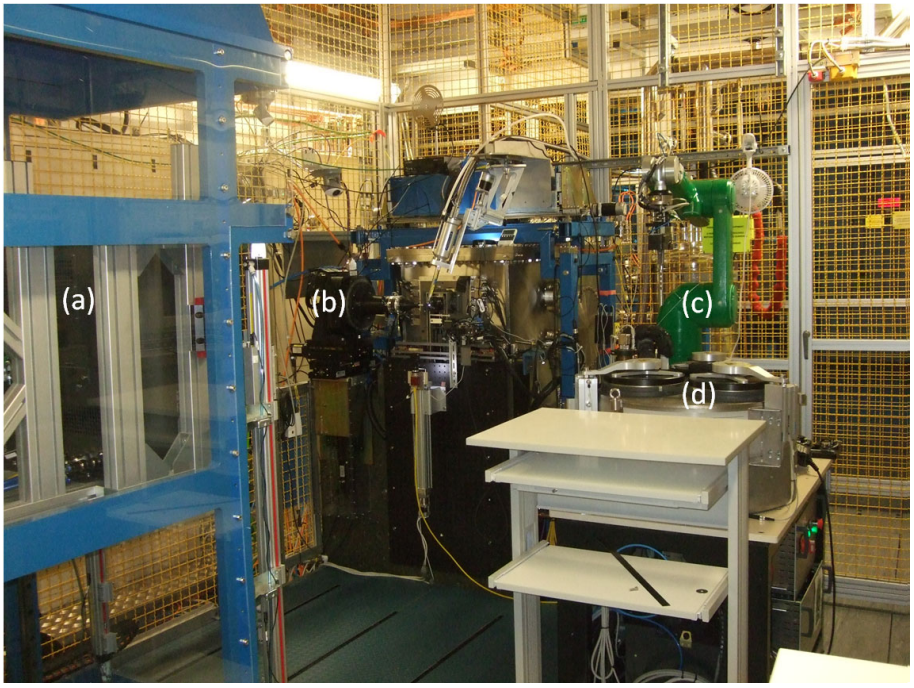

Fig. 4. The end-station for the macromolecular crystallography beamline I24: (a) Pilatus3 6M detector; (b) goniometer stage; (c) CATS robotic sample changer; (d) sample dewar.

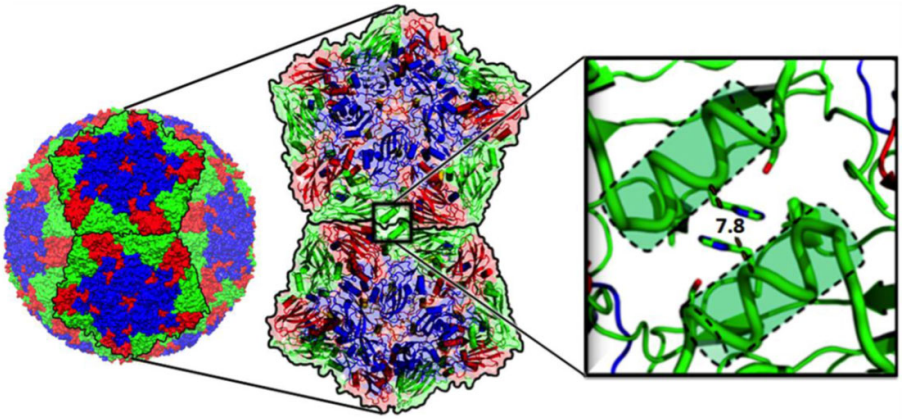

Fig. 5. The crystal structure of FMDV serotype A22 highlighting the His 93 residues mutated in the stabilised capsid.

The formation of this bond greatly increases the stability of the viral capsid, a key consideration in production and distribution of vaccine [7].

2.3 Beamline I23, a long-wavelength macromolecular crystallography beamline to allow the measurement of the anomalous signals from the sulphur or phosphorous atoms, present in native protein or RNA/DNA crystals, to be determined for phasing

The long-wavelength macromolecular beamline I23 is entering the final stages of its commissioning and construction. It is a facility specifically designed to operate in the 8.3 keV to 3.1 keV (1.5 Å to 4.0 Å) energy range which will allow the small anomalous signals from the sulphur and phosphorous atoms that are present in native protein or RNA/DNA crystals to be measured. The beamline uses a U27 in vacuum undulator as its X-ray source and the optics are fairly conventional. A Si(1 1 1) double-crystal monochromator(which was designed and assembled in-house from components fabricated by a number of suppliers [8]) is used for energy selection and this passes the beam to two pairs of horizontally deflecting mirrors. The first mirror pair provides focusing and this is achieved by initially using a cylindrical mirror to focus the beam in the vertical followed by a bent flat mirror to provide horizontal focusing. Downstream of this first

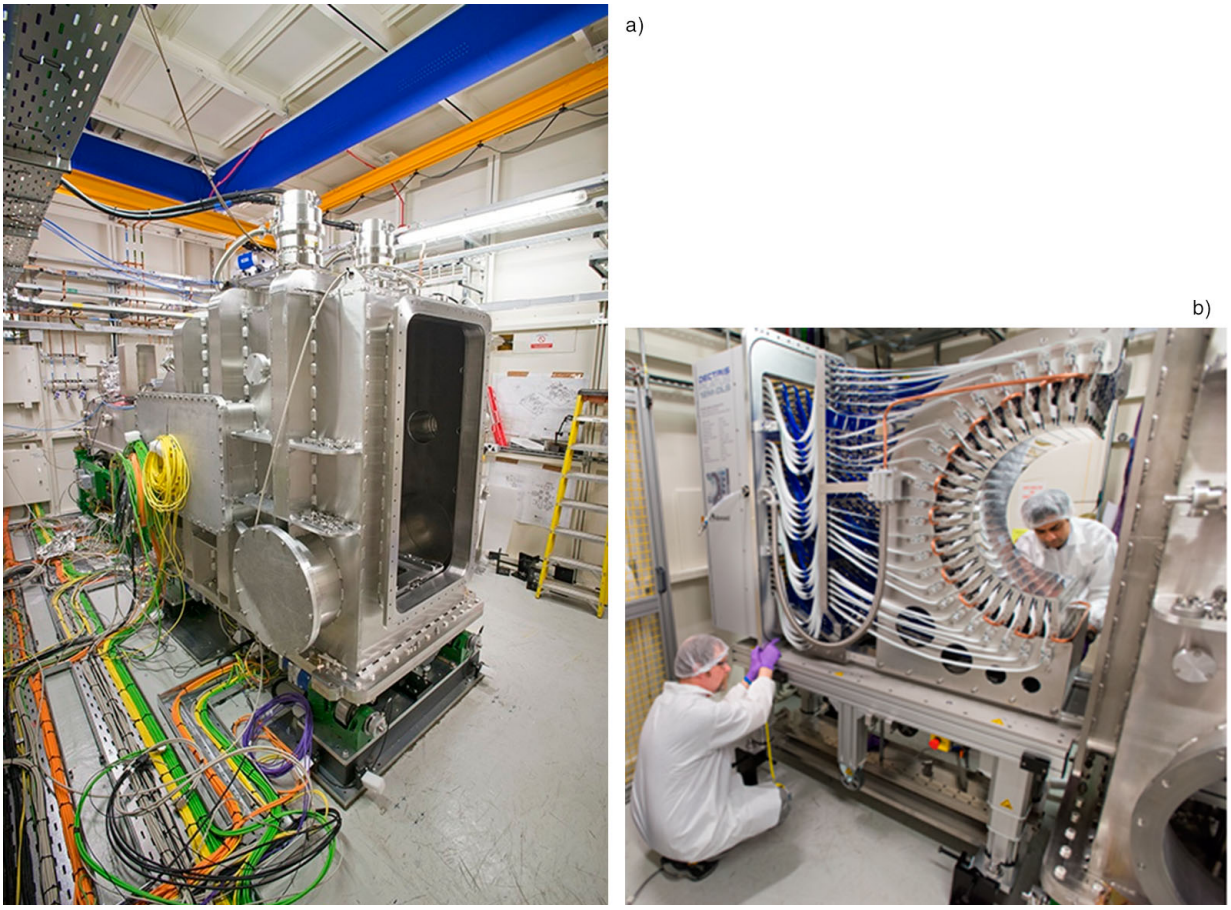

Fig. 6. (a) The vacuum vessel for the I23 end-station before the installation of the instrumentation. (b) The Pilatus 12M detector of beamline I23 being installed into the end-station vacuum vessel.

set of mirrors is a pair of horizontally deflecting flat mirrors with stripes of Si, Ni and Pt, for harmonic rejection. The mirror assembly was manufactured by FMB Oxford incorporating optical elements from SESO (focusing mirrors) and InSync (harmonic rejecting mirrors). The combination of the four mirrors allows the beam to emerge from the mirror vessels following its original, undeviated, path from monochromator. Therefore, for those tomography studies where the effects of harmonics are not an issue, the mirrors can be rapidly withdrawn to provide a large beam at the sample position, straight from the monochromator, with no realignment of the end-station required. A large unfocussed, harmonic-free, beam can also be provided for tomography by withdrawing only the pair of focusing mirrors but some realignment of the end-station and harmonic rejection mirrors is then required.

Due to the low photon energies the entire end-station is mounted within a vacuum vessel to eliminate the effects of absorption of the diffracted beams in air, fig. 6a. The vessel occupies a substantial fraction of the experimental hutch. At the upstream end of the vessel are the X-ray collimation stages and sample viewing optics along with a port for an X-ray fluorescence detector. To one side of the vessel is the goniometer. Initially this will be a single-axis instrument but, once initial commissioning has been completed, this will be replaced by a kappa goniometer. As the goniometer will be in vacuum the sample cannot be cooled using the usual open-flow nitrogen cryostat but instead the sample is cooled conductively via the goniometer. The goniometer is cooled via a pulse tube cooler which connects to the instrumentation through a port at the top of the vacuum vessel. Sample changes are carried out via an air-lock and manipulator mechanism positioned on the opposite side of the vacuum vessel from the goniometer. The samples are also kept cool during their storage and transfer to the goniometer while they are under vacuum. At the downstream end of the vessel is a Pilatus 12M detector, fig. 6b, for shutterless data collections. The 120 detector modules are within the vacuum [9] and are held in a frame to provide a half cylinder surface with a radius of 250 mm and a width of 424 mm. The detector can be translated along the beam direction by 250 mm but once the centre of the radius is placed at the sample position the 2θ angular coverage is $\pm 100^\circ$ (40.3° laterally). The read out electronics are in air, outside the vacuum vessel, and they are connected to the detector modules via a set of electrical feed-throughs in a large bulkhead, which also allows the whole detector to be inserted into the vessel at installation.

Table 1. Key parameters and instrumentation for the macromolecular crystallography beamlines at Diamond Light Source. The beam sizes are quoted with the horizontal dimension first and the vertical dimension second (FWHM).

Beamline	I02	I03	I04	I04-1	I24	I23
Type	tuneable (5–25 keV)			optimised fixed 13.5 keV	tuneable microfocus (7–20 keV)	tuneable long wavelength (3–12 keV)
Beamsize μm FWHM, h × v	70 × 20	70 × 20	70 × 20	60 × 40	5 × 5 to 50 × 50	variable TBD
Flux (ph/s) 10^{-4} bandpass	10^{12}			10^{11}	$> 10^{12}$	TBD
Pilatus detector	P6M-F (25 Hz)			P2M (30 Hz)	P3-6M (100 Hz)	P12M (10 Hz) (half cylinder)
Sample changer	Rigaku			CATS	CATS	in-house design
Containment	CL1	Cl3	CL1	CL1	CL2	–
Humidity control	On request (all beamlines and offline)					–
Multi-axis gonio	–	–	Mini kappa	Mini Kappa	In development	In development
Minibeam aperatures	yes	yes	yes	yes	In development	In development
Crystallisation plates	soon	yes	soon	yes	yes	–
Crystal washer	yes	yes	soon	yes	yes	–
Remote access	All beamlines at all times					In development

At the time of writing the X-ray optics, beam collimation stages, sample viewing optics, the preliminary single-axis goniometer and the detector are being commissioned and the sample changer is being made ready for installation. The first user experiments are scheduled for early 2015.

All of the macromolecular crystallography beamlines use EPICS and GDA software for beamline control and data collection while ISPyB (Information System for Protein Crystallography Beamline: ISPyB is a joint development between Diamond Light Source, the ESRF, BM14 (e-HTPX) and the EU funded SPINE project) is used for the tracking of samples, the cataloguing of data and metadata, the reporting of outcomes, and data archiving [10].

The key parameters for all of the macromolecular crystallography beamlines at Diamond are summarised in table 1.

3 The physical sciences beamlines

3.1 Beamline I19, a high-flux tunable-wavelength facility for the study of small-molecule systems using single-crystal diffraction techniques

Of all the physical sciences beamlines at Diamond the one that, perhaps, bears the closest comparison to those in the life sciences is I19, the small-molecule single-crystal diffraction beamline, as it uses essentially the same methodologies for data collection and structure determination [11]. However, as small-molecule systems tend to diffract to higher resolution, the end-station instrumentation on I19 is quite different to that on the macromolecular crystallography beamlines. Beamline I19 uses a 2 m *in-vacuum* undulator with a 21 mm period as its X-ray source and its optics hutch is broadly similar to those of I02, I03 and I04 in that it contains a double-crystal monochromator with a cryocooled Si(1 1 1) crystal set and a pair of bimorph mirrors to focus the beam in the horizontal and the vertical planes. The monochromator was manufactured by Bruker (formerly Accel) while the mechanical components for the mirrors, including the vacuum vessel, were manufactured by FMB Oxford which house the Seso supplied optical elements. The mirrors, which have a 3 mrad incidence angle, are also coated with Pt and Rh stripes to offer the same harmonic-free energy windows as the MX beamlines. As will be discussed in more detail later, I19 has two experiments hutches, experiments hutch 1 (EH1) and experiments hutch 2 (EH2), in tandem and as a consequence the mirrors are required to focus the monochromatic beam at sample positions that are 38 m and 44 m from the source, respectively. The focussed beam has an approximately Gaussian profile with dimensions of 90 μm horizontally (h) × 60 μm vertically (v)

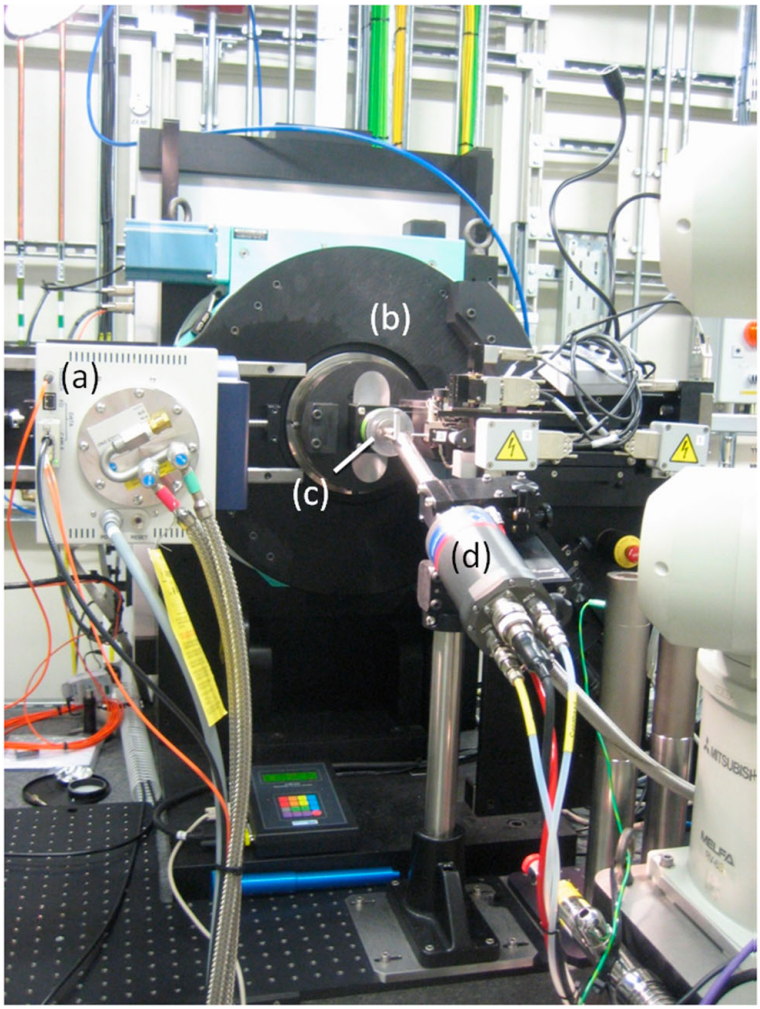

Fig. 7. The 4-circle kappa-geometry diffractometer housed in EH of beamline I19: (a) Saturn 724+ CCD detector; (b) diffractometer; (c) microglide motorised goniometer head; (d) cryostream.

FWHM at the first sample position in experiments hutch 1 (EH1) and 185 μm (h) $\times$ 130 μm (v) FWHM at the second sample position in experiments hutch 2 (EH2). The mirrors are routinely defocused to provide a larger beam size for the majority of experiments in EH1 (defocused beam size is 170 μm (h) $\times$ 85 μm (v) FWHM) as this provides greater flexibility on sample size (ideally, for small-molecule crystallography, the entire sample is bathed in the primary beam). The approximate Gaussian profile is closely maintained after this slight defocusing with minimal structure due to residual slope error in the mirrors.

The experiments hutches both contain a kappa geometry four-circle diffractometer which provide the multi-axis high-resolution data collections that small-molecule crystallography demands. The diffractometer housed in EH1 was manufactured by Crystal Logic and Rigaku integrated it with their Saturn 724+ CCD detector along with the same type of sample changing robot as used on I02, I03 and I04. Rigaku also incorporated an Oceaneering 3-axis "microglide" motorised goniometer head so that crystals can be centred on the diffractometer remotely once they have been mounted, or exchanged, by the robot, figs. 7 and 8. The diffractometer is used in this mode for most chemical crystallography studies, although the microglide can be readily swapped for a standard IUCr goniometer head for some sample environment studies —such as high-pressure experiments with diamond-anvil cells or *in situ* studies of gas-exchange in porous materials using a gas cell. The instrument has a sphere of confusion on the order of 20 μm with the microglide attached and approximately 10 μm with a conventional goniometer head. The EH1 diffractometer and robotic sample changer are controlled by the Rigaku CrystalClear and Cameraman Windows software and data analysis can be performed, also in Windows, via CrystalClear, Agilent's CrysAlisPro or Bruker's Apex-II software. Most sample environment studies, however, are carried out on the much larger Newport diffractometer in EH2, fig. 9. The Newport diffractometer is capable of carrying sample environments weighing up to 25 kg at its sample position, while maintaining a 75 μm sphere of confusion, such as a closed-cycle cryostat, and is better suited to more demanding studies. The location of EH2 downstream of EH1 allows users to prepare for complex experiments while EH1 is still in use.

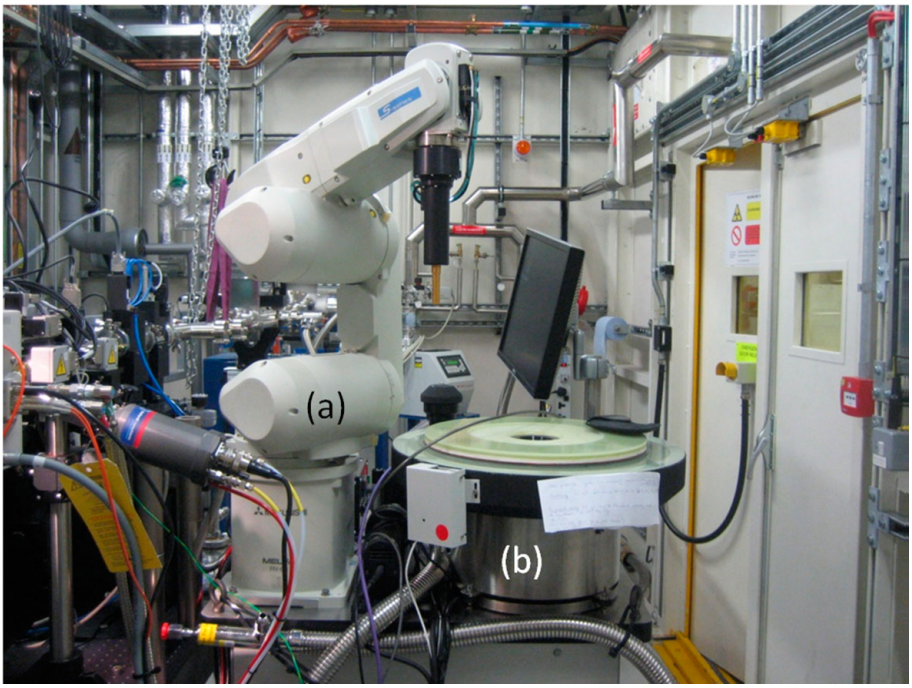

Fig. 8. The robotic sample changer housed in EH1 of beamline I19: (a) robot arm; (b) sample dewar.

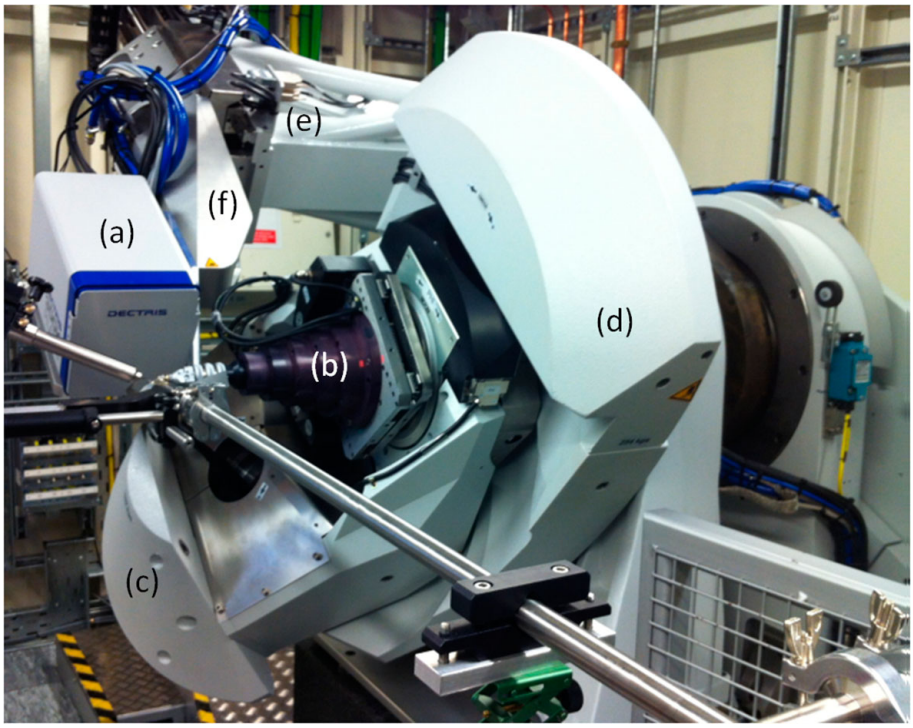

Fig. 9. The Newport 4-circle kappa-geometry diffractometer housed in EH2 of beamline I19: (a) Pilatus 300K detector; (b) phi axis and integral x, y, z sample stage; (c) kappa arm; (d) omega circle; (e) 2Theta arm; (f) detector translation stage.

For pump-probe time resolved studies, this can prove to be invaluable as the laser and optics can be installed and optimised well ahead of the experiment while user operation can still be maintained in EH1. The Newport diffractometer can be used with either an Agilent Atlas CCD detector or a Pilatus 300K detector. The latter is now being used routinely for shutterless data collections. This has transformed the capabilities of the beamline as many studies, particularly those requiring the gas cell, can be carried out extremely rapidly. The Pilatus 300K detector is also capable of carrying out time-resolved studies without recourse to the mechanical chopper, which is also situated in EH2, and data collections exploiting either the triggering or gating modes of the detector can allow very sophisticated timing sequences to be carried out relatively easily: whether they involve different time delays to be determined simultaneously within a single data collection or they allow the encoding of the Hadamard pattern on to a sequence of images. The gating mode of the Pilatus 300K detector can also operate at the orbital frequency of the storage ring and allow the isolated bunch of either single bunch mode or hybrid mode to be isolated. This allows time domains in the pico-second range to be studied. When the Pilatus 300K detector is in use, diffractometer control is carried out via EPICs and the GDA while for experiments requiring the Atlas CCD detector data collections are carried out with Windows software supplied by Agilent and Newport. Data analysis is currently carried out with CrysAlisPro for both detectors.

In the next few months the diffractometer in EH1 will be upgraded to allow both faster data collections and the study of crystals that are smaller, and more weakly scattering. The new goniometer will have the fixed-chi geometry and it will have a sphere of confusion of approximately 3 microns. A Pilatus 2M detector will also be used that can be operated at a larger sample to detector distance, to reduce the background from air-scatter, and as it operates in shutter-less mode it will also allow significantly faster data collections to be carried out. We will also take the opportunity to improve the sample viewing system and harmonize the data collection software with the robot and the rest of the beamline.

An example of the complex chemical crystallography that can be undertaken in EH1 of beamline I19 is demonstrated by the work of Argent *et al.* [12], where they determined the structure of an unusual high-nuclearity metal-organic nanosphere. The synthesis of high-nuclearity transition metal co-ordination clusters has been the focus of substantial research over recent years due to interest in tailoring the properties of the nanoscale objects. Such molecules are a target for bottom-up nanotechnology fabrication techniques and, despite many potential applications, large clusters are often most admired for their attractive architectures and topologies that are reminiscent of Platonic and Archimedean solids, and even the capsids of viruses [13]. In this example, the authors report the synthesis and full structural characterisation of a highly unusual Cd_{66} cluster —a high-nuclearity system which is composed of a central inorganic Cd-oxo-hydroxy-nitrate core surrounded by an organic shell of ligands. This highly unusual molecular compound has a molecular weight of about 23,800 Daltons, has an external diameter of 3.18 nm, and an internal diameter of 1.22 nm, and therefore, can be considered to be a metal-organic nanosphere, fig. 10.

3.2 Beamline I16, a single-crystal diffraction facility for the study of the magnetic structure and subtle structural features in a diverse range of materials

I16 (Materials & Magnetism) was the first crystallography beamline in operation at Diamond, as it was one of the earliest in the phase-I build, and it continues to serve a vibrant user community in the fields of condensed matter (electronic ordering, magnetism) and X-ray physics [14]. It is also a natural choice for carrying out novel experiments where no dedicated facility exists. From the early years of magnetic scattering, it was apparent that high-flux high-resolution resonant scattering facilities can probe a wide variety of fascinating phenomena in X-ray physics, including resonant and non-resonant magnetic scattering, anisotropic resonant scattering —often going beyond the dipole approximation— and studies of minute atomic displacements in strain waves. Most of these phenomena exhibit complex behaviour with respect to beam energy, azimuthal rotation and photon polarization, and so I16 was designed with such studies in mind. The beamline combines very high flux with no comprise in resolution with a focal spot size, using the original optics, down to $20 \times 185\,\mu$m (FWHM). The beamline uses an in-vacuum U27 undulator as its X-ray source and the beam is first monochromated, by a cryocooled, channel-cut, Si(1 1 1) monochromator, manufactured by FMB Oxford, before it passes through a diamond phase retarder, which provides control over the polarization of the beam. The photon energy can be tuned between 3.3 keV to 15 keV for most experiments, although a 2.7 to 3.0 keV energy window can be made available on request using a non-conventional setup of the optics. Once monochromated, the beam is focused in the vertical by a horizontally-deflecting cylindrical mirror before it is focused in the horizontal by a bent flat mirror which also deflects the beam in the horizontal parallel to its original path. The mirror mechanics were manufactured by Bruker (formerly Accel) and the optical elements were fabricated by Seso. The flat mirror has two stripes, a plane Si part and a Rh-coated part, for harmonic rejection. The beam is further conditioned by a series of slits before it is passed on to the experiments hutch.

The rather commodious experiments hutch of I16 houses a Newport 6-circle diffractometer, fig. 11. The diffractometer has the necessary additional degrees of freedom, over a more conventional 4-circle diffractometer, to allow direct azimuthal scans of reflections to be performed, and it has a 50–70 μm sphere of confusion at the sample position. The capability of performing this type of scan is crucial for the accurate determination of magnetic structure.

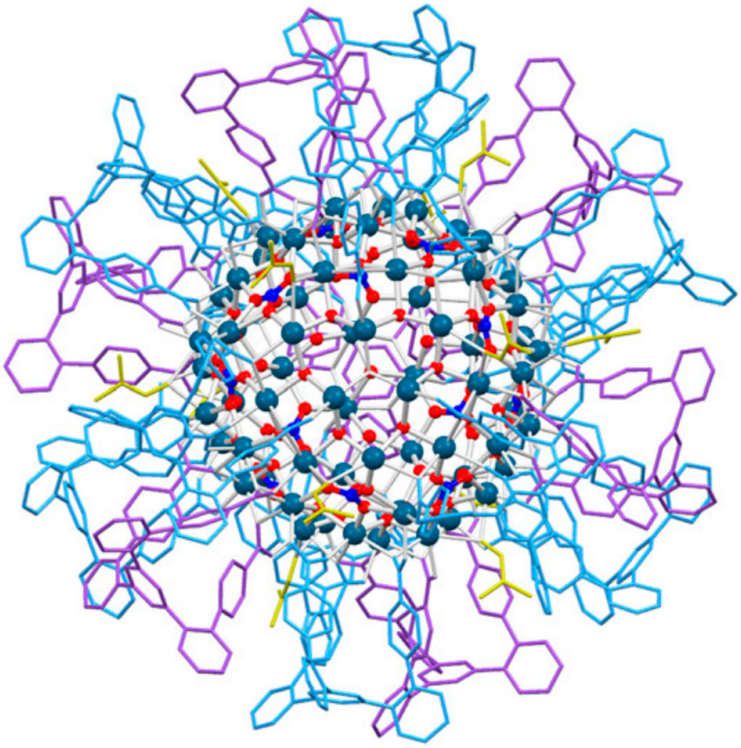

Fig. 10. View of the Cd66 nanosphere. Atom colours: cadmium dark blue; oxygen red; ligands of different symmetry coloured light blue and purple. Ligand at front of ball removed to aid clarity.

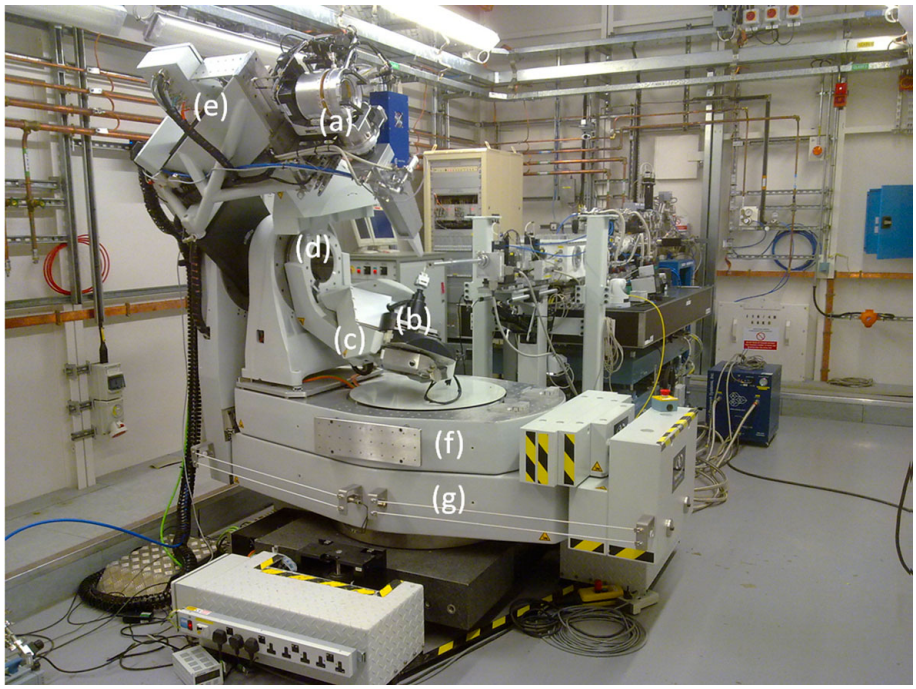

Fig. 11. The 6-circle kappa-geometry Newport diffractometer of beamline I16: (a) detector and analyser crystal stage; (b) phi axis and integral x, y, z sample stage; (c) kappa arm; (d) Theta circle; (e) 2Theta arm; (f) mu circle; (g) gamma circle.

The 2Theta arm supports a variety of detectors and these are arranged, rather like the fingers of a hand, on a rotary stage to allow a specific detector to be selected for a particular measurement. Additionally, the detector stage can also be rotated along an axis parallel to the 2Theta arm, rather like a wrist (to continue with the hand analogy), so that the analyser crystal can additionally probe the polarization of the beam diffracted by the sample. For a more rapid surveying of reciprocal space, the detector arm also holds a Pilatus 100K area detector, which is now used in shutterless mode for the majority of experiments that the beamline supports. The beamline is also equipped with Pilatus 2M detector which is mounted to the diffractometer on a separate, detachable, platform. When this detector is in operation the 2Theta arm is oriented and fixed in the vertical position. The diffractometer is controlled via EPICS and the GDA while data analysis is mainly undertaken using MATLAB.

Since first user operation in 2007, the beamline optics, detectors and sample environments have been enhanced significantly. As mentioned earlier, the low energy range has been extended down to 2.7 keV, providing access to several very important atomic resonances such as the Ru L-edges. A pair of silicon harmonic rejection mirrors provides extremely high harmonic rejection, and new KB mirrors can focus the entire monochromatic undulator beam to a $2 \times 6\,\mu$m spot (FWHM) at the sample (smaller with reduced flux). The beamline has a wide range of detectors and, as well as those already mentioned, it has a Medipix detector, which can be operated in shutterless mode. Sample environment options also now include a sub-2K cooler.

As experiments become increasingly demanding in terms complexity and sample size, continuing development of the facility is essential. Currently work is being carried out on more versatile phase-plate polarizer and linear polarization analyser systems, as well as a miniature sample manipulator to enhance the beamline's capability for studying very small ($\sim$ 10 micron) sample crystals, thus paving the way to allow the study of materials that have not been accessible in the past.

Of the many forms of magnetic material that are studied on beamline I16, multiferroics have gained a lot of recent interest. These materials possess both ferromegnetic properties, meaning they can form permanent magnets, and ferroelectric properties, where the materials possess spontaneous electric polarization, and this rare combination of characteristics makes them especially interesting from both purely academic and technological standpoints. At present, many technologies such as computer hard drives and security access cards rely on magnetic materials containing coded information. The magnetic material will have this information 'written' onto it via electric induction, a process that requires a lot of energy. The use of a multiferroic material would allow the information to be written using an electric field, a process which would be more energy efficient.

Typically, materials tend only to be either ferromagnetic or ferroelectric, and those that are multiferroics only exhibit these properties at low temperatures. The study of the prototypical multiferroic $BiFeO_3$ by Johnson *et al.* [15] was aimed at understanding the underlying physics governing the magnetic behaviour of this material. They used I16 to image the magnetic domains at the surface of a ferroelectric monodomain in a single crystal of $BiFeO_3$. Magnetic domains on the order of several hundred microns were observed which corresponded to cycloidal modulations of the magnetization along the wave vector $k = (\delta, \delta, 0)$ and symmetry equivalent directions. The rotation direction of the magnetization in all magnetic domains, determined by diffraction of circularly polarized light, was found to be unique and in agreement with predictions of a combined approach based on a spin-model complemented by relativistic density-functional simulations. Imaging of the surface shows that the largest adjacent domains display a 120° vortex structure, see fig. 12.

3.3 Beamline I11, a high resolution powder-diffraction beamline for the structure determination of complex materials under non-ambient, time-resolved, and long duration conditions

The high-resolution powder-diffraction beamline, I11, was built in phase-II of beamline construction and it has been operational since early 2008 [16]. It uses an in-vacuum U22 undulator as its source and a cryocooled double-crystal monochromator, manufactured by Bruker (formerly Accel), selects photon energies in the range of 6 to 25 keV. The monochromatic beam is passed to a pair of flat vertically deflecting mirrors, which provide harmonic rejection via Pt, Rh or Si stripes. The mirror mechanics were manufactured by Irelec while mirrors themselves were produced by Crystal Scientific. The beam size at the sample position is ideal for powder-diffraction, $\sim$ 0.6 mm (v) $\times$ 2.5 mm (h) (FWHM). The experiments hutch houses a heavy-duty Newport 3-circle powder-diffractometer (2δ, 2θ and ω), fig. 13, with a sphere of confusion at the sample position of 12 μm. A range of non-ambient cells, weighing up to 25 kg, can be mounted in the sample circle (θ). Mounted radially on the upper segment of the 2θ-circle are five high-resolution detector modules each composed of 9 detector elements, with the individual elements each containing their own multi-analyser crystal (MAC). Occupying the lower segment of the diffractometer is the 90° position-sensitive detector (PSD) built using Mythen2 silicon modules for time-resolved studies. The PSD is mounted on the δ-circle and is concentric with the 2θ-circle, and allows the PSD to be scanned separately from the MAC. Although the PSD has a more limited resolution than the MAC ($\Delta\delta \sim 0.04°$, for a 0.5 mm capillary, compared to $\Delta 2\theta \sim 0.002°$), it is possible to collect complete powder-diffraction patterns in less than 1 second in a shutterless mode. The PSD can be used in combination with the MAC if required. Apart from the diffractometer itself, there is a large robotic sample changer (the arm can

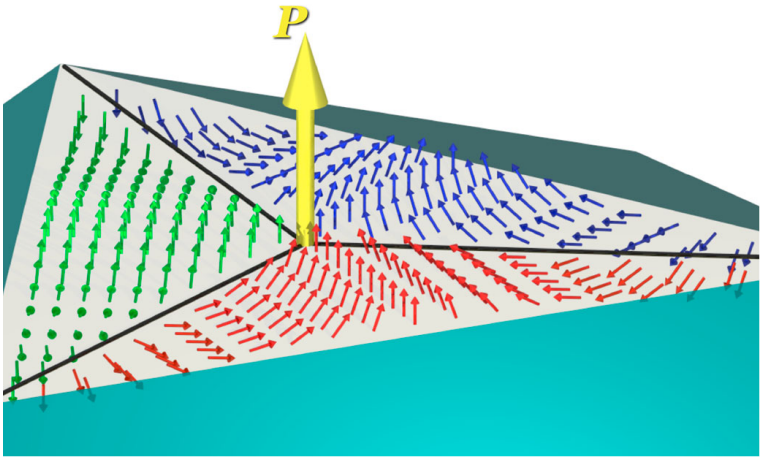

Fig. 12. Illustration of the three magnetic domains of $BiFeO_3$, coloured red, blue and green, imaged using non-resonant magnetic diffraction techniques at I16. The magnetic moments (spins) in each domain rotate in a common plane, and with a common sense of rotation determined by the direction of the electric polarisation, shown by the yellow arrow.

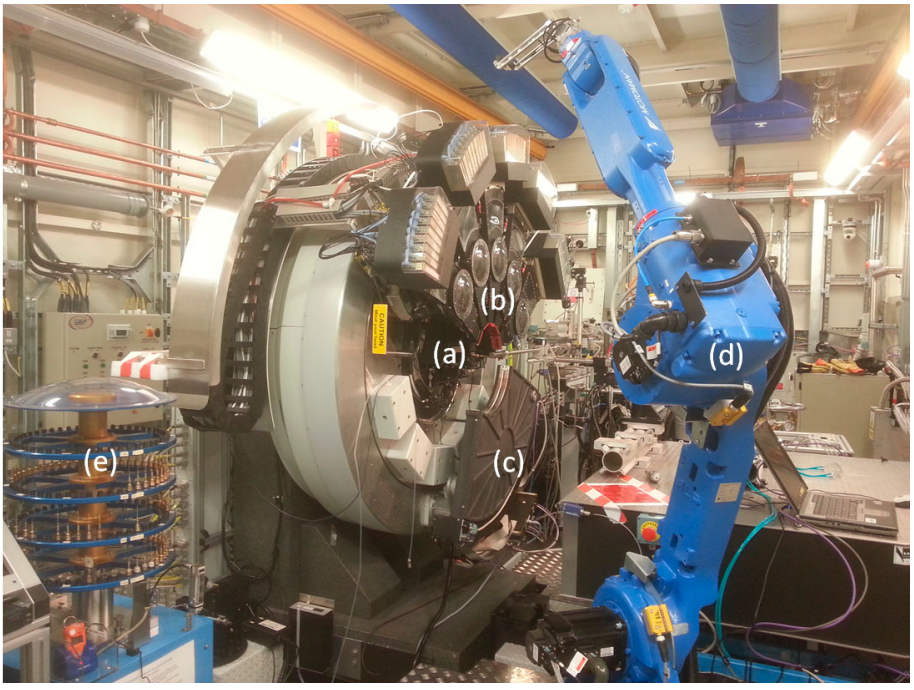

Fig. 13. The high-resolution powder-diffraction diffractometer and robotic sample changer housed in EH1 of beamline I11: (a) sample stage; (b) MAC high-resolution detectors; (c) high-speed PSD detector; (d) robotic sample changer; (e) sample carousel.

extend to over 2 metres) with its associated carousel, which can hold up to 200 samples contained in capillaries. This has been operating reliably since the beamline became operational. Recently, an upgrade project has been completed to achieve micro-focussed beam. A set of compound refractive lenses on an alignment table in the experimental hutch has been commissioned to provide small beam sizes ranging from 10 to 100 μm over the 15–25 keV energy range. This additional capability is particularly useful for studies of single micro-polycrystalline particles with nano-domains or for experiments which require a small beam for high spatial resolution. In this mode an optical table is placed downstream of the diffractometer which supports an area detector and a range of motorised stages for the sample holder and a series of collimators and pin-holes.

Within the last year I11 has undergone a radical change with the construction of a second experiments hutch, where the control room used to be, downstream of the unaltered first experiments hutch. A new control room has been built on the side of the beamline. The new experiments hutch (EH2) will provide a facility for long duration experiments (LDE) to complement the existing capabilities of I11 for high-resolution and fast (millisecond)time-resolved studies,

Fig. 14. The long duration experiments hutch (EH2) of beamline I11 during the final stages of equipment installation: (a) Pixium area detector; (b) detector translation stage; (c) sample stage.

fig. 14. The LDE facility will open up new opportunities for those experiments which require weeks to months of periodically monitoring "slow" changes. It will be of particular benefit to certain research areas such as batteries and fuel cells where important information on the development of phases over time cannot be obtained via *ex situ* methods. Other research areas that will similarly benefit include studies of slow crystallisation, long term stability of gas storage materials, mineral evolution due to seasonal effects, thermal and electrical power cycling, and corrosion science. The hutch contains a very large granite table upon which a number of experimental cells can be mounted. The cells are placed on their own individual set of linear stages so that each cell, in turn, can be placed in the beam and powder-diffraction data collected. The table has individual stages for 2 large cells, 4 medium sized cells and 18 smaller cells, while the hotel for the robotic sample changer can hold up to 30 sample environment cells that can be placed on a separate stage on the table. The data are recorded on a Pixium area detector which is mounted on a translation stage that allows the adjustment of the sample to detector distance to suit the requirements for each of the sample positions. The distance can be adjusted in the range from 40 cm to 400 cm, although the full detector distance is reliant on the position of the sample stage on the table: *i.e.* only the stage at the upstream end of the table has the full detector range available to it. Once set up and running, the LDEs are left on their sample stages and automated data collections are carried out periodically over the period of weeks or several months. A range of sample environments such as battery cells, heating stages and high pressure gas cells can be also be accommodated for user requirements.

The beamline is controlled via EPICS and the GDA and data analysis is carried out via Fit2D, for area-detector data, and a range of refinement packages including GSAS and TOPAS.

The high-resolution capabilities of I11 now make it possible to determine the relatively large and complex structures with powder-diffraction methods that were previously only accessible via single-crystal diffraction techniques. The structure of the phosphonate metal-organic framework (MOF) system STA-16(Co) [17], which was determined from powder-diffraction data collected on I11, is shown in fig. 15. MOFs are promising porous materials for the storage of gases and pharmaceutical agents and they can be compared in the crystalline state with pore sizes that can greatly exceed those of aluminosilicate zeolites. Their architecture is also extremely flexible as the crystal structure, and pore size, can be readily controlled via the choice of metal and the design of the linker molecule. With the trend for increasing pore size, and the expectation that this will be accompanied by an ever greater storage capacity, the structures of MOFs are steadily becoming more complex. The determination of these structures is certainly beyond the capabilities of laboratory-based X-ray powder-diffraction instrumentation. The work of Wharmby *et al.* demonstrates, however, that the structure solution of these complex MOFS are easily within the reach of a state-of-the art powder-diffraction beamline, such as I11, and will also allow the *in situ* study of the gas-exchange within these materials, which is a crucial element in the understanding of their behaviour and the optimisation of their function.

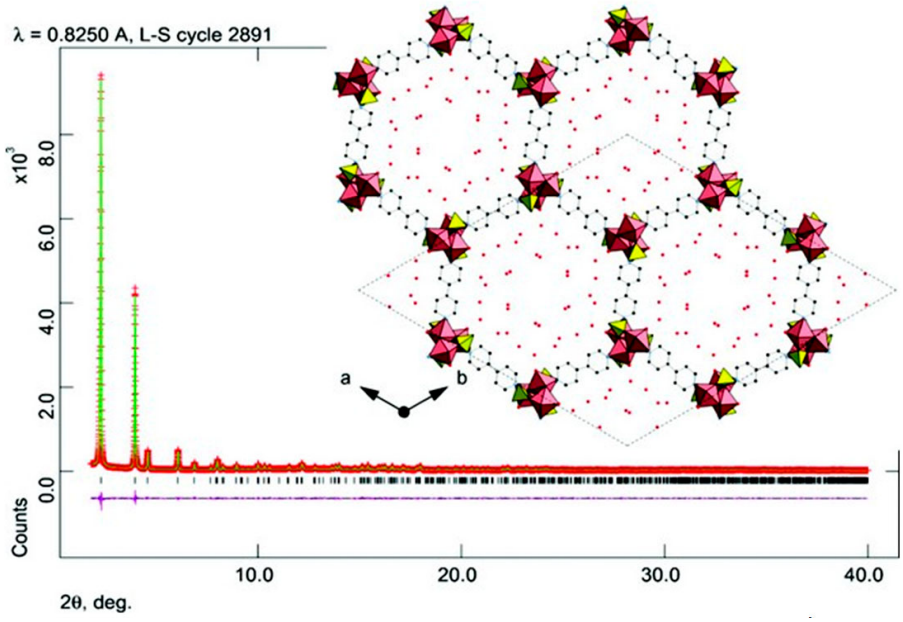

Fig. 15. Rietveld fit to the high-resolution powder-diffraction data from I11, $\lambda = 0.825028$ Å, with structure of STA-16(Co) overlain. Hydrogen atoms not shown. (R-3, $a = b = 41.2870(2)$ Å, $c = 6.2630(1)$ Å; $R_{\rm wp} = 0.0467$, $R_p = 0.0338$).

3.4 Beamline I15, a high-energy beamline for studying materials under extreme conditions, at high-pressures and high- or low-temperatures, using either single-crystal or powder-diffraction techniques

The Extreme Conditions Beamline, I15, came into operation in July 2007 and is one of Diamond's Phase I beamlines. It is a facility for the study of materials at extreme conditions, *e.g.*, pressures and temperatures and, as such, it uses the high-energy X-rays generated by a superconducting wiggler as its source. The white beam can be used directly for experiments but, generally, the beam is monochromated by a Si(1 1 1) double-crystal monochromator, which selects photon energies in the 20–80 keV energy range. The beam is then focused in the horizontal and the vertical by a pair of mechanically bent KB mirrors onto two possible sample positions within the experiments hutch (EH1). The horizontally focussing mirror also provides harmonic rejection. The monochromator and the mirror assembly were supplied by Instrument Design Technology (IDT). EH1 is equipped with a 6-circle Newport diffractometer (Station 1), see fig. 16, in the upstream position and a second table at the downstream position (Station 2). The 6-circle diffractometer, which is very similar to that on I16 and has a 50 μm sphere of confusion, can support a variety of area detectors including: a MAR345 image plate system; a Perkin Elmer flat panel detector, an Agilent Atlas CCD detector and a shutterless Pilatus 100K detector. The latter two can be mounted on the diffractometer arm for single-crystal diffraction studies. The focused beam size at this station is $\sim 70\,\mu$m (FWHM) but smaller beam sizes (down to 20 μm) can be achieved by collimation. On station 2 a pair of miniature KB mirrors, of 300 mm optical length, focusses the beam down to about 10 μm. The MAR345 image plate and the Perkin flat panel detector are used to record the powder pattern. The microfocusing capabilities are required for high-pressure studies in the Mbar region and for laser-heating experiments. The latter will employ two fibre YAG lasers (each having a power-output of 100 Watt and a wavelength $\lambda = 1090$ nm) for heating the sample, which is contained in the pressure cell, from both sides. Temperature measurement will be carried out via the collection of the thermal emission spectra over the spectral range from 500 nm to 900 nm.

The beamline is controlled via the standard EPICS and GDA software layers while data analysis is undertaken using 2D data reduction with the Data Analysis WorkbeNch (DAWN) [18,19], for powder-diffraction data collected with the area detectors, and for single-crystal diffraction data Agilent's CrysAlisPro Windows-based software is used in conjunction with the Atlas CCD detector.

The ongoing construction of the side-station on I15, which is dedicated to X-ray pair-distribution studies, will widen the diffraction capabilities of I15. This branch line will use a fraction of the wide horizontal X-ray fan produced by the wiggler. All beamline components such as primary slits, diagnostics, monochromator and mirror will be installed in the existing optics hutch. The horizontally-deflecting bent Laue monochromator will produce horizontally focused X-rays at energies of 40, 65, and 76 keV. A bi-morph multilayer mirror will be used for the vertical focusing. A monochromated beam with variable size (minimum is about 700 μm (h) and 20 μm (v) FWHM) will be available at the sample position in the new experiments hutch (EH3). Two large 2-D area detectors will be available to collect

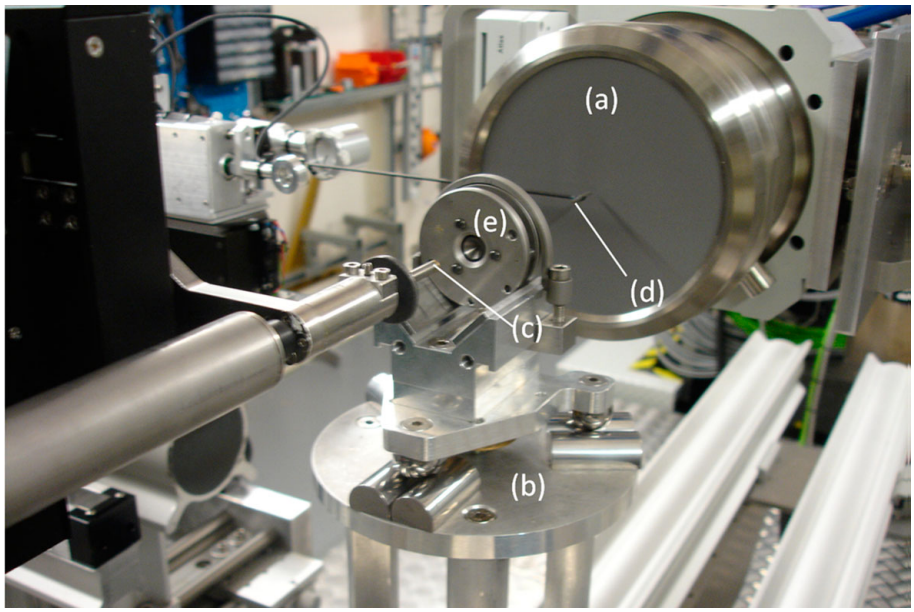

Fig. 16. A close up view of the 6-circle kappa-geometry Newport diffractometer (Station 1) housed inside EH1 of beamline I15. In the configuration shown in the figure, the diffractometer is set up for single-crystal diffraction with the Agilent Atlas CCD detector: (a) CCD detector; (b) kinematic sample stage; (c) pinhole collimator stage; (d) beamstop; (e) diamond-anvil cell.

scattering data over a wide Q range. Their distance to the sample will be tuneable which allows the reciprocal space resolution and maximum Q coverage to be optimised. For the Bragg data collection one detector can be moved to a position of about 1m downstream the sample. This new beamline will be available for users in 2016.

Beamline I15 allows the investigation of the high-pressure structural behaviour of materials to be studied rapidly via powder-diffraction and an investigation of the phase behaviour of the iridium-hydrogen system (iridium in a hydrogen medium) up to 125 GPa, by Scheler *et al.* [20] is a noteable example of this type of work. At 55 GPa an additional phase appears in XRD patterns of iridium. The new phase can easily be indexed in the simple cubic $Pm\text{-}3m$ space group and exhibits a significantly larger volume per atom than pure iridium at the same pressure. This indicates the formation of a compound material, *i.e.*, a metal hydride (as opposed to a phase transition in pure iridium). The bulk modulus was also found to be 190 GPa indicating that the material is significantly more compressible than pure Ir, which has a bulk modulus of 383 GPa. *Ab initio* calculations show that the hydrogen atoms occupy the face-centered positions in the metal matrix, making this the first known noninterstitial noble metal hydride and, with a stoichiometry of IrH_3, the one with the highest volumetric hydrogen content. Computations also reveal that several energetically competing phases exist, which can all be seen as having distorted simple cubic lattices. Slow kinetics during decomposition at pressures as low as 6 GPa suggest that this material is metastable at ambient pressure and low temperatures.

The principal parameters for all of the physical crystallography beamlines at Diamond are summarised in table 2.

4 Peripheral laboratories

All of the macromolecular crystallography beamlines have a sample preparation area adjacent to the experiments hutch for their users. The area is used mainly for the handling of sample pucks from their transport storage Dewars for transfer into the Dewar of the robotic sample changer. The beamlines are also equipped with microscopes in this area to assist in the occasional mounting of individual crystals when the need arises. The beamlines also share access to a development laboratory and a second laboratory, that is use is split between technique development and user support. This laboratory hosts offline humidity control and microspectroscopy equipment and a Xe pressure cell for crystal derivatisation, all of which are bookable in advance. Apart from the membrane protein crystallography laboratory, which is situated in the experimental hall at Diamond, all of the current molecular and biochemistry related research activities are now carried out in the laboratory space in the Research Complex at Harwell.

The peripheral laboratories for the physical science beamlines are generally quite different from one another as they are required to support a wider range of scientific activities. As the general work flow for small-molecule crystallography is broadly similar to that for macromolecular crystallography, the support facilities for beamline I19 are, perhaps, the most reminiscent to those for the MX beamlines. I19 also has a sample preparation area adjacent to the control room that allows users to prepare their samples (often a large number of samples) for their beamtime. The preparation

Table 2. Key parameters and instrumentation for the physical crystallography beamlines at Diamond Light Source. The beam sizes are quoted with the horizontal dimension first and the vertical dimension second (FWHM). The beam diameter can be reduced to the values in parenthesis through the use of a series of (a) refractive lenses or (b) pinholes. The values for the flux are quoted for: a bandpass of 10^{-4} at (c) 15 keV and (d) at 6 keV; and for a bandpass of 10^{-3} at (e) 25 keV and (f) at 30 keV.

Beamline	I19		I16	I11		I15	
Type	Tuneable (5–25 keV)		Tuneable (2.7–3.0 keV and 3.3–15 keV)	Tuneable (6 to 25 keV)		Tuneable (20–80 keV)	
	EH1	EH2		EH1	EH2	Station 1	Station 2
Beamsize μm FWHM, h × v	90 × 60	185 × 130	185 × 20	2500 × 600 (10 × 10)[a]	500 × 200	70 × 70 (20 × 20)[b]	10 × 10
Flux (ph/s)	10^{12} c	10^{12} c	10^{13} d	10^{13} c	10^{12} e	10^{12} f	10^{12} f
Detectors	Saturn 724+ CCD	Pilatus 300K, Atlas CCD	Pilatus 100K (main), Pilatus 2M	MAC, Mythen2 PSD	Pixium	Atlas CCD, Mar 345, Pilatus 100K, Perkin Elmer 1621 EN	
Sample changer	Rigaku	–	–	Motoman	Motoman	–	–
Diffractometer	4-circle κ CrystalLogic	4-circle κ Newport	6-circle κ Newport	3-circle powder Newport	Modular stages	6-circle κ Newport	Hexapod goniometer

area offers users two powerful microscopes for crystal assessment and mounting, along with a Schlenk line for the handling of air-sensitive samples. EH1 is also equipped with its own sample preparation bench, which has dedicated microscope and a Schlenk line. This setup is useful for extremely air-sensitive samples that require a rapid transfer to the diffractometer once the sample has been mounted. To maintain sample quality during the mounting process the sample preparation area in EH1 is also supplied with an X-Temp2 device [21] which maintains sample crystals under a flow of cold nitrogen gas while they are viewed and mounted under a microscope. As well as the facilities on the beamline, I19 also has a peripheral laboratory that is equipped with an additional sample preparation microscope, a fume cupboard and a Braun glovebox, which has its own microscope. The lab also contains facilities for the preparation of diamond-anvil cells, such as a spark eroder for gasket hole drilling and a ruby fluorescence spectrometer for the measurement of pressure. The lab also has a dedicated cryostream and gas-cell control system for the preparation of samples for gas-cell studies prior to beamtime. Although the I19 peripheral laboratory is not set up for any involved synthetic chemistry work, it is equipped with balances, glassware and a drying oven that users may find useful to assist with sample preparation and re-crystallisation.

On beamline I16, as each experiment is expected to last several hours if not a few days, the beamline does not have a dedicated sample preparation area as the sample throughput is concomitantly low. The samples are prepared in the I16 peripheral laboratory where they are usually bonded to copper mounts that are subsequently attached to the cold-finger of a cryostat. The lab is equipped with two microscopes to assist with this work. The lab also has a fume cupboard, for instances where solvents are used in sample preparation, and there is additional ancillary equipment, such as hot plates and glassware.

The experiments hutches EH1 and EH2 on the powder-diffraction beamline, I11, each have their own very generously proportioned control cabin. The latter also has a proportion of space set aside for the preparation of sample cells for the long duration experiments. Additionally, the beamline has two peripheral labs that are available to users of both hutches. The sample preparation lab is primarily available to users so that they can load their samples into capillaries and sample holders prior to, or during, their beamtime. The lab is supplied with basic equipment, such as pestles and mortars, glassware and balances, to make these tasks as straightforward as possible. Microscopes and a fume cupboard, for solvent use, are also available. The second laboratory is used for offline analysis and it is equipped with Raman, IR and UV spectrometers, a low-resolution SEM and furnaces for non- or low-hazard materials synthesis.

Aside from the control room and optics and experimental hutches there are also several further laboratories associated with the I15 beamline. A diamond-anvil cell sample preparation laboratory at the end of the beamline equipped with several microscopes, several systems for gasket hole drilling, and a ruby fluorescence spectrometer. A second laboratory houses the equipment for off-line diamond-anvil cell laser heating with both YAG and CO_2 lasers and the spectrometers for thermal emission measurements. As well as laboratory space on the beamline I15 also has peripheral laboratories in the experimental hall: a gas-loading apparatus, which allows diamond-anvil cells to be pre-loaded with gas pressure media at up to 1.5 kbar initial pressure; a Horiba Raman system for on- (via fibre) and off-line Raman spectroscopy experiments with diamond-anvil cells; space for off-line cryostat experiments; and a preparation laboratory for Paris-Edinburgh large-volume high pressure experiments. Further down the experimental hall is also a glove-box with a microscope available that is suitable for loading diamond-anvil cells.

It should be noted that, although the users of a beamline take priority for access to its particular laboratory, all of the peripheral laboratories at Diamond are available on request. This allows valuable pieces of equipment, such as spectrometers, to be generally available to all users of Diamond even in the event that the equipment is in another beamline's laboratory.

5 Conclusions

The revolution in detector technology and the on-going development of beamline optics and precision goniometry, make these exciting times for crystallography and structural science at synchrotron facilities such as Diamond. These developments not only allow us to collect data at higher speed, with greater sensitivity on ever smaller and more weakly diffracting crystals but they also allow us to perform new types of experiment, particularly where time resolution, ranging from microseconds to months, is required. With these developments there is an ongoing and ever more pressing need to manage the large volumes of data that can be produced and provide not only an automated means of processing the data, from the raw images to a final electron density map, but to also archive all of the raw data and the accompanying meta data in a robust and sustainable way.

With greater data collection speeds, there is an accompanying requirement for the more rapid handling of samples. For macromolecular crystallography, the use of robotic sample changers have become a crucial element of the data collection pipeline and the handling, mounting, storage and transportation of crystals is now completely geared towards the use of robots on the beamlines. In parallel with these developments for conventional studies using pin-mounted samples, there has also been a rapid development of *in situ* studies for macromolecular crystallography where structures have been determined from samples held within their original crystallisation trays. With these developments, the productivity of MX beamlines at Diamond will continue to increase along with their ability to handle ever more challenging samples. In the Physical Sciences the beamlines continue to adapt to the growing and diversifying requirements of the users and, as for the MX beamlines, this has often led to profound changes not only to instrumentation but also to the layout and construction of the beamlines themselves.

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References

1. E.M.H. Duke *et al.*, AIP Conf. Proc. **1234**, 165 (2010).
2. E.M.H. Duke, L.N. Johnson, Proc. R. Soc. A **466**, 3421 (2010) doi:10.1098/rspa.2010.0448.
3. F. Cipriani, F. Felisaz, L. Launer, J.-S. Aksoy, H. Casserotto, S. Cusack, M. Dallery, F. di-Chiaro, M. Guijarro, J. Huet, S. Larsen, M. Lentini, J. McCarthy, S. McSweeney, R. Ravelli, M. Renier, C. Taffut, A. Thompson, G.A. Leonard, M.A. Walsh, Acta Cryst. D **62**, 1251 (2006).
4. J. Ohana, L. Jacquamet, J. Joly, A. Bertoni, P. Taunier, L. Michel, P. Charrault, M. Pirocchi, P. Carpentier, F. Borel, R. Kahn, J.-L. Ferrer, J. Appl. Cryst. **37**, 72 (2004).
5. M. Micco, G.W. Collie, A.G. Dale, S.A. Ohnmacht, I. Pazitna, M. Gunaratnam, A.P. Reszka, S.J. Neidle, Med. Chem. **7**, 2959 (2013).
6. G. Evans, D. Axford, R.L. Owen, Acta Cryst. D **67**, 261 (2011).
7. C. Porta, A. Kotecha, A. Burman, T. Jackson, J. Ren, S. Loureiro, I.M. Jones, E.E. Fry, D.I. Stuart, B. Charleston, PLoS Pathog **9**, e1003255 (2013).
8. J. Kelly, S. Alcock, H. Patel, J. Phys.: Conf. Ser. **425**, 052009 (2013).
9. J. Marchal, A. Wagner, Nucl. Instrum. Methods Phys. Res. A **633**, S121 (2011).
10. S. Delagenière, P. Brenchereau, L. Launer, A.W. Ashton, R. Leal, S. Veyrier, J. Gabadinho, E.J. Gordon, S.D. Jones, K.E. Levik, S.M. McSweeney, S. Monaco, M. Nanao, D. Spruce, O. Svensson, M.A. Walsh, G.A. Leonard, Bioinformatics **27**, 3186 (2011).
11. H. Nowell, S.A. Barnett, K.E. Christensen, S.J. Teat, D.R. Allan, J. Synchrotron Rad. **19**, 435 (2012).
12. S. Argent, A. Greenaway, M. del Carmen Gimenez-Lopez, W. Lewis, H. Nowell, A.N. Khlobystov, N. Champness, M. Schroder, A.J. Blake, J. Am. Chem. Soc. **134**, 55 (2012).
13. M.D. Ward, Chem. Commun. **30**, 4487 (2009).
14. S.P. Collins, A. Bombardi, A.R. Marshall, J.H Williams, G. Barlow, A.G. Day, M.R. Pearson, R.J. Woolloscroft, R.D. Walton, G. Beutier, G. Nisbet, AIP Conf. Proc. **1234**, 303 (2009).
15. R.D. Johnson, P. Barone, A. Bombardi, R.J Bean, S. Picozzi, P.G. Radaelli, Y.S. Oh, S.W. Cheong, L.C. Chapon, Phys. Rev. Lett. **110**, 217206 (2013).

16. S.P. Thompson, J.E. Parker, J. Potter, T.P. Hill, A. Birt, T.M. Cobb, F. Yuan, C.C. Tang, Rev. Sci. Instrum. **80**, 075107 (2009).
17. M.T. Wharmby, J.P.S. Mowat, S.P. Thompson, P.A. Wright, J. Am. Chem. Soc. **133**, 1266 (2011).
18. M. Basham, J. Filik, M.T. Wharmby, P.C.Y. Chang, B. El Kassaby, M. Gerring, A.W. Ashton, *Data Analysis WorkbeNch (DAWN)*, to be published in J. Synchr. Radiat. (2015).
19. DAWN, Data Analysis WorkbeNch, `http://www.dawnsci.org/`.
20. T. Scheler, M. Marqués, Z. Konôpková, C.L. Guillaume, R.T. Howie, E. Gregoryanz, Phys. Rev. Lett. **111**, 215503 (2013).
21. T. Kottke, D. Stalke, J. Appl. Cryst. **26**, 615 (1993).