National Research Facility Annual Reporting Template

1. Background:

EPSRC National Research Facilities are research facilities that provide resources to the Engineering and Physical Sciences research community on a scale where there is limited availability in the UK for reasons such as:

- The relative cost of the equipment;
- Dedicated kit in every University is not needed;
- Particular expertise is needed to operate the kit or interpret the results;
- Progress is enhanced by sharing information or software.

The EPSRC National Research Facilities are mainly supported through 5 year contracts and underpin research across the EPS landscape.

A regular process for the submission and assessment of National Research Facility Statements of Need has been put in place which provides research communities with the opportunity to suggest new National Research Facilities. Statements of Need for newly proposed facilities are then assessed and tensioned against the Statements of Need submitted by existing facilities whose contracts are coming to an end.

2. Annual Reporting Process

During the 5 year period for each facility, EPSRC wishes to support the facilities in providing an optimal service to their users. To assist with this EPSRC has an annual reporting process for the National Research Facilities where the performances of the existing National Research Facilities are assessed by an experienced panel which includes representatives from the EPSRC Capital Equipment Strategic Advisory Team:

As part of the assessment process this annual reporting template has been provided and this year must be filled in by **all** National Research Facilities that have an ongoing arrangement with EPSRC. The relevant EPSRC managers will also be invited to provide a short summary of their interactions and experience with the facility.

After the panel meeting, the assessment of the panel will be fed back to the facilities to enable them, together with their steering committee and EPSRC contact, to ensure the best possible service is provided to the user community.

Timeline 2019:

- Reporting Period for this Annual Report: **1st September 2018 – 31st August 2019**
- Deadline for Annual Reports**: 12st November 2019**
- Assessment by Panel**: December 2019**
- Feedback to Facilities: **January 2020**

Annual Report for EPSRC National Research Facilities

Facility: XMaS

Address: ESRF - The European Synchrotron, 71, avenue des Martyrs, 38000 Grenoble, France

Director: Prof. Chris Lucas (University of Liverpool) and Dr. Tom Hase (University of Warwick)

Beamline Responsible: Dr. Didier Wermeille (University of Liverpool)

Description of the Facility (max. 1/2 page): Please give a brief description of the facility and its main objectives for a non-specialist audience.

The XMaS National Research Facility is a synchrotron x-ray beamline embedded in the heart of the European Photon and Neutron (EPN) Science Campus in Grenoble, France and is managed by the Universities of Liverpool and Warwick. The beamline is part of the European Synchrotron Radiation Facility (ESRF), which forms part of the EPN campus along with the other European institutes including the European Molecular Biology Laboratory (EMBL) and the Institut Laué-Langevin (ILL) neutron source. The original beamline was conceived primarily as a tool to study magnetic materials, hence the acronym **X**-ray **Ma**gnetic **S**cattering (XMaS). The beamline has been supporting users since 1997 but has a far broader remit than magnetic scattering. It supports active research groups in over 40 UK universities (representing over 400 independent researchers) and covers research in materials science, physics, chemistry, soft condensed matter as well as biomaterials and healthcare. The facility also supports international collaboration; attracting additional users from ~30 international institutions. The facility is an enabling tool serving the materials science community (including academic researchers, national research laboratories and industry) – hence its rebirth as the UK's **X**-ray **Ma**terial **S**cience beamline at the ESRF. It plays a major role in underpinning interdisciplinary projects and contributes directly to societal challenges such as energy storage and recovery, the digital economy and advances in healthcare technologies as well as contributing to the UK research infrastructure.

The main objectives of the facility are to provide access to the UK materials science community to a state-of-the-art x-ray facility (source and experimental equipment) and to provide training for early career scientists and both postgraduate and undergraduate students in advanced scientific methodologies. These activities take place within a vibrant international environment.

Contract/Grant Period and Costs

Term: 5-years
Start Date: 15th November 2018
End Date: 14th November 2023

Total cost: £6,909,720: split between Liverpool (£3,427,966) and Warwick (£3,481,754)
Total capital cost: £2,039,674: split between Liverpool (£1,151,772) and Warwick (£887,902)

Spend on track? **YES**

Grant references: University of Liverpool EP/S020802/1 and University of Warwick EP/S020845/1

Key Performance Indicators (KPIs) and Service Level (SLs) (max. 3 pages):

Since Dec. 2018 the ESRF has been upgrading its magnetic lattice (EBS upgrade programme) and has been shut down. User operation is expected to resume in August 2020. This NRF reporting period spans the ESRF shutdown. During this period the XMaS beamline is undergoing a refurbishment and rebuild of its infrastructure to align to the new ESRF lattice. In this section we report on User statistics over the lifetime of the facility as a NRF (formerly MRF) to provide an overview of user statistics.

Key Performance Indicators (KPIs)

A) The Number of Individual Researchers and University Research Groups [“users”] that have made use of the XMaS beamline in that Period. This should be expressed as a Total Number for that period.
B) Number of User Complaints received during the period. This should be expressed as a percentage of the Total Number of User Approvals made within the period.
C) The Uptime of the beamline within the period. This should be expressed as a percentage of the Total Available Time within that Period.
D) The Number of research outputs. This should be expressed as a Total Number for the period.

Service Level Agreements (SLs)

- Requests for beamtime will have decisions made within 20 days of the PRP meeting subject to knowledge of the ESRF review process. In 'exceptional' cases Users will be informed by the service operator if these benchmark times are going to be exceeded and an explanation provided.
- Facility Users will have access to facility staff for assistance on site.
- The facility will be operational and available for use for 80% (eighty percent) of the maximum possible operational time.
- The facility will train all new Users in the safe and effective use of the beamline.
- The facility will perform a minimum of 2 (two) publicity activities per year.
- The facility will generate a minimum of 15 (fifteen) research outputs per year.
- The facility will respond to all User enquiries clearly and quickly in line within 5 (five) working days for emails and 2 (two) working days for telephone enquiries.
- The facility will respond to User complaints within 10 (ten) working days.
- The facility will treat all proposals equally, fairly and in confidence.
- The facility will treat all Users equally and fairly.
- The facility will uphold high standards of integrity in all operations and in contact with Users.

Over the lifetime of the facility, all KPIs and SLs have been exceeded. A list of current quarter KPIs as well as full historical data is maintained on our webpage (www.xmas.ac.uk) and is updated quarterly. Access to the facility is governed by a Collaborating Research Group (CRG) contract with the ESRF. This stipulates that 30% of the full flux beamtime is allocated through the ESRF panels in a worldwide call. This time can be accessed by UK researchers and for completeness we include these visits in our reporting statistics. The numbers specific to this access route are reported below in parentheses.

A) The number of Individual Researchers and University Research Groups visits [2012-2019]:
There have been **632 (184)** individual researcher visits from a cumulative total of **242 (29)** UK and **61 (70)** International research groups visits since XMaS has been funded as a mid-range facility. We report these numbers similarly to DIAMOND and the ESRF where each use of the facility is classed as a separate visit. The breakdown by operational quarter is shown in Figure 1. The data show consistent metrics, with the number of researcher visits, training and uptime consistent across the operational periods. We train, on average, 25 new users, students and PDRAs per quarter. The metrics clearly show that the user community continues to evolve and grow.

B) Number of user complaints [2012-2019]: XMaS has received zero (0) complaints over its lifetime of >20 years.

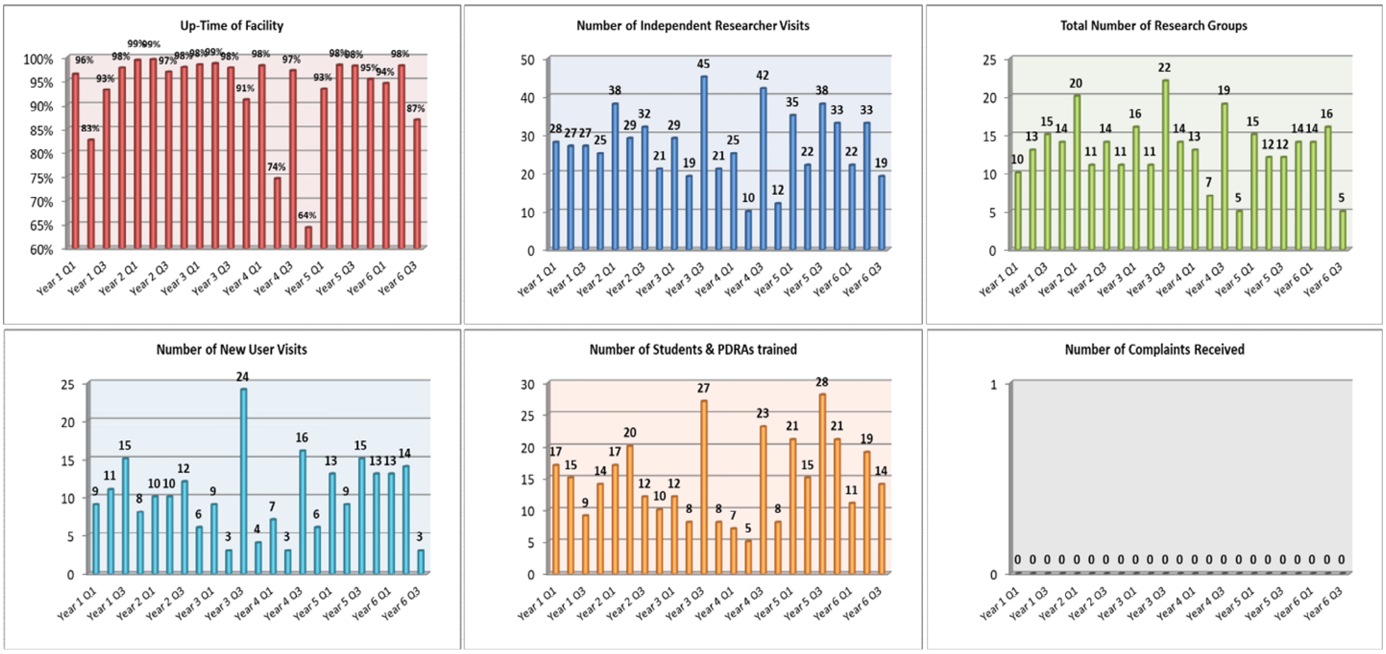


Figure 1: KPIs as a function of facility time displayed by operational quarter.

Historical metrics

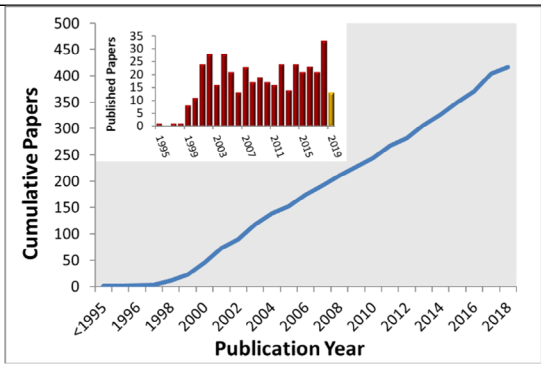
As a mid-range facility, XMaS has successfully increased both the number of users and experiments that we have been able to accommodate (Figure 2). This has been due to increasing use of 9 shift experiments and efficiency gains in terms of configuring the beamline and sample environments. The data show throughput increases in both user visits and the number of experiments performed for the same number of scheduled operational hours.

Figure 2: Historical performance metrics for XMaS including the number of scheduled hours (left), the number of user visits to the facility (centre) and the number of individual experiments performed on the beamline (right).

C) Uptime of the beamline between 2012 and 2019:

The uptime of the facility during the past six years has been 94.6%, consistently above the contractually required 80%. We have chosen an internal expectation of 95% uptime based on the historical operating uptime. For many operational periods, this was exceeded, but some independent and unforeseen events had a deleterious impact on the overall average. These included a design flaw in a commercial detector and a failure of the He line to a Joule-Thomson cryostat, both necessitating a cancellation of an experiment. Commissioning of the cryogenic monochromator experienced delays (Q16) which resulted in the postponement of 1 week of beamtime in Q17. We also had a failure of the safety critical beam-shutter. Outside of these unfortunate instances the downtime is a combination of facility instrumentation failures, user sample problems and external causes including failure of the synchrotron storage ring over which we have no control. Operational resilience has been increased by updating detectors and re-designing the beamline shutter to have a mean time between failure of >10 years. In the 2 month period prior to the ESRF shutdown, essential commissioning of the cryogenic monochromator and identification of upgrade pathways were performed to ensure that XMaS user operations will recommence as soon as the ESRF resumes user operations in August 2020.

D) The number of research outputs: The total number of XMaS papers has now exceeded 400. Over the reporting period (2018-19) there were a total of 23 published papers building on a record 2018 in which 33 papers were published. Although publication rates are relatively consistent there has been a steady shift to output in high impact journals (Figure 3). In 2018, nearly a third of the output was in high-impact journals; here defined as those journals with an impact factor >7 but also including APL and Scientific reports. Approximately 70% of the papers are co-authored with facility staff. There are also many conference and seminar presentations which are more difficult to capture. As noted in our last quinquennial ESRF review, the publication record is comparable to, or even exceeds that, of similar beamlines at both the ESRF and DIAMOND. The research output of XMaS is rated highly by the ESRF management with XMaS work selected as "ESRF Highlights"; 2012(1), 2013(2), 2014(3), 2015(2), 2016(2), 2017(1) and 2018(2). A further 6 XMaS articles have appeared in the ESRF newsletter, 7 as ESRF spotlights and 2 in their general news. The director of research, Harald Reichert, states "*It is my pleasure to confirm that XMaS contributes substantially, and on a competitive basis to the scientific output of the ESRF.*" Prof. Andy Dent (Diamond) states "*on similar beamlines at Diamond, which usually have 1 week long experiments, the typical publication rate is somewhere between 12 and 20 high quality publications per annum. Often they are complex requiring more than one visit in order to merit a publication in a top journal.*"



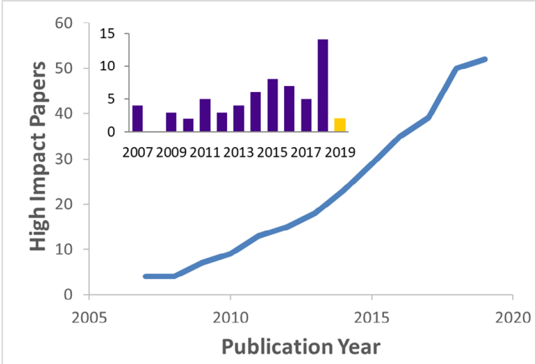


Figure 3: Top: Cumulative outputs of all peer reviewed publications. Inset, annual output as of 1st Oct. 2019. Bottom: Same plot but for high impact papers from 2007.

Access Requests

Although not a designated KPI of the facility, the demand for the facility can be gleaned from the user application data. Unlike most beamlines, XMaS schedules all of the operational modes of the ESRF, including the 16- and 4-bunch modes. Thus, the maximum possible time is allocated to users through a peer review panel which ensures equality, transparency and fairness in the allocation process. Our access statistics are therefore skewed downwards when compared with other beamlines which (a) limit user operations to high flux modes and (b) reserve time for in-house research. Subscription rates are determined from the ratio of the total number of requested shifts and the total number of shifts delivered for user operations. The average subscription for the review period was 1.64 for CRG time and 2.73 for ESRF allocations. If we were to only use the multi-bunch beam slots in the calculation of subscription rates (as is done for the ESRF statistics) the subscription rate is ~2.4. The delivered subscription rate also masks efficiency improvements delivered over recent years. Although demand has increased by 30-40%, we have managed to accommodate more users and keep the subscription rate constant.

Offline Facilities

In addition to the synchrotron beamline, XMaS has developed a range of "offline" facilities exploiting previous capital investments. These include a magneto-transport laboratory for material characterisation under electric and magnetic fields without x-rays, as well as a micro-focus x-ray source for diffraction, reflectometry and simple SAXS experiments. Access to the facilities was disrupted during the hutch extension work in 2018 and limited availability through 2019 as staff resources were needed for beamline developments, but over the reporting period **8** proposals were received with **12** user visits covering **186 days** of operations for the x-ray machine. For the characterisation lab, a further **2** proposals with **2** user visits and **51** operational days were received. These statistics are in line with historical averages from 2015. Of the total possible shifts, the x-ray facility operated at about 30% capacity with the characterisation lab at 10%. Any increase in these loads must be weighed against increasing impact on staff resource whose primary responsibility remains with the operation of the beamline itself.

Users (max. 2 pages): For the reporting period please provide information and data on the users of the facility over the reporting period.

Since Dec. 2018 the ESRF has been upgrading its magnetic lattice (EBS upgrade programme) and has been shut down. User operation is expected to resume in August 2020. This NRF reporting period spans the ESRF shutdown. During this period the XMaS beamline is undergoing a refurbishment and rebuild of its infrastructure to align to the EBS upgrade. In this section we report on User access over the lifetime of the facility as a NRF (formerly MRF) to provide an overview of user engagement.

User statistics are derived from the data gleaned from the ESRF access protocols and by independent user feedback provided at the end of each experimental session. As part of the contract with the ESRF we provide 30% of the available beamtime for public access. The KPI data returned below refers to the 70% facility time reserved for UK-led proposals with the numbers in parenthesis describing the ESRF publicly allocated time.

Summary of Key User statistics [2012-2019]:
Number of user visits: **632 (184)** from **303 (99)** user groups visits**;** number of new users: **233 (93)**; student visits: **255 (78)** and PDRA visits: **86 (32)**. Of these user visits 6% were from non-academic institutions including Diamond Light Source, AWE Aldermaston, the Swiss Light Source and the EU Institute for transuranic studies. Figure 4 shows a map of the user groups from across the UK. We have also received proposals from the Warwick Manufacturing Group as part of the advanced materials catapult. We are currently exploring partnerships with the catalysis hub at RAL, the nuclear decommissioning authority, the US Navy, Electrosciences Ltd. and the Surrey 5G centre. Proprietary beamtime has been delivered for Johnson Matthey.

Figure 4: Geographical spread of our UK users and their collaborators. Red markers correspond to user groups since 2012.

Third Party Activities [2012-2019]:
XMaS was part of the recently completed EMRP funded *Nanostrain Project* (2014-2016) which explored multiferroic materials under applied electric fields with cycling rates of up to 1 MHz. The collaboration, led by the National Physical Laboratory (NPL), brought together several of the EU metrology labs along with industrial partners including IBM (New York) to explore strain mediated transistor devices to replace current CMOS technology and overcome the current energy crisis in Si-based devices. XMaS is now part of the *Advent Project* (2018-2020), defining the metrology for advanced energy-saving technology in next generation electronic applications. *Advent* builds upon the success of *Nanostrain* and the key objectives are to provide an *in situ* time resolved electric field capacity (4 MV/cm) to explore frequencies up to 1 MHz and magnetic field up to 3 Tesla whilst also exploring the temperature dependence of these materials. Another key objective is to apply *in-operando* stress (200 MPa) to ferroelectric samples and to integrate this to the beamline control system. The main partners in *Advent* are Physikalisch-Technische Bundesanstalt, Germany (PTB) and other EU metrology laboratories. We are actively seeking third-party engagement in two new EU funded programmes; "JRP g-10 **OpMetEnergy** - Operando Metrology for Energy Storage Materials" and "JRP g-19, Ageing and Fatigue of Energy Materials". Both programmes build on metrologies developed with our users and will expand the operational capabilities for all users of the beamline. The EMRP activities continue to be highlighted in several trade magazines and other online resources. Many of our users work with third parties, recent examples include British Petroleum and Siemens. We now ask the users to give specific details in their end of run surveys on such engagement activities. Direct industrial engagement activities have occurred with Johnson Matthey.

User feedback [2012-2019]:

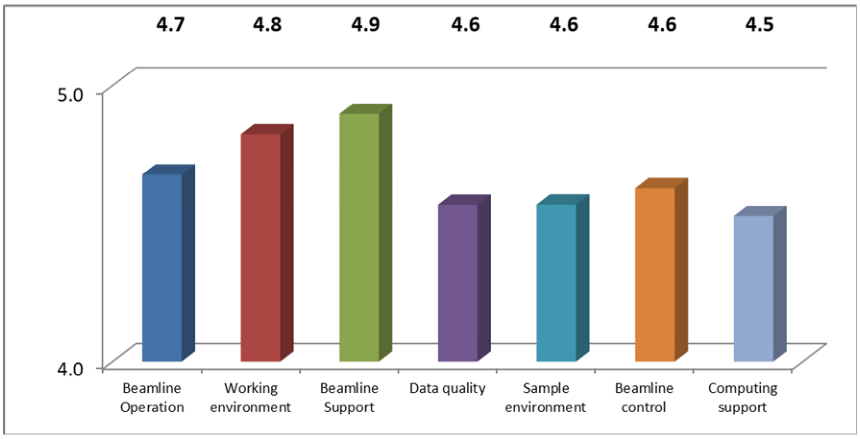


Figure 5: User feedback on facility operations covering the period 2012-2019.

End of run surveys returned by our users provide additional information about performance. These metrics, which are summarised in Figure 5 for the period 2012-2019, are scored out of 5, with 5 being the highest score. Overall the data are very positive. Efforts to make the beamline controls more efficient and to develop clearer data analysis pathways have been identified as desirable areas for improvement and progress on these issues is being made during the current upgrade period (during the ESRF shutdown).

User Research Areas [2012-2019]:

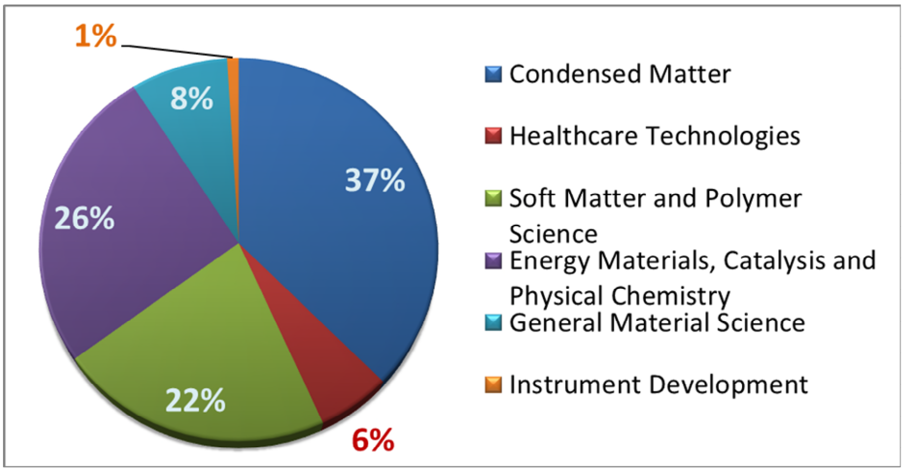


Figure 6: XMaS experiments by research area as reported by our users for the total mid-range facility time.

The science supported on XMaS covers a much broader range of challenges than are normally found on any single SR beamline. Due to the multidisciplinary user community, which is stretched across Physics, Chemistry and Materials disciplines within the UK science base, there is a strong mapping to many of the science and technology areas that underpin EPSRC's remit. The user research profile continues to evolve, as seen in Figure 6, with new research areas, such as energy materials, strengthening during this period. XMaS remains in a unique position whereby the best practices of one community have been shared, adapted and implemented by others, to the benefit of all users, and wider afield through our international collaborations.

Scientific Excellence (max. 2 pages)

XMaS has a long history of engaging with recurrent and new users to continually expand the scientific remit. Being open to user-driven developments is at the heart of our operational ethos and enables existing metrologies to be improved and new scientific areas to be studied. Most recently, we have explored the use of wavelength-dispersive spectroscopic methods, extending future capabilities to include High Energy Resolution Fluorescence Detection (HERFD), X-ray Absorption Spectroscopy (XAS), X-ray Emission Spectroscopy (XES) and Resonant Inelastic X-ray Scattering (RIXS). Efficiency gains through bespoke sample environment and beamline configurations continued in parallel with developments in multi-modal analyses from several data channels and at frequencies up to 1 MHz. A new beamline paper summarising the current status was published as part of the celebration of 50 years of SR in the UK (Phil. Trans. R. Soc. A 377, 20180237, pp 12 (2019)). In terms of scientific excellence, a sub-set of the current research activities from the 2018-20 period are presented below, grouped around challenge themes:

Energy research: Research covers photovoltaics, battery materials and catalysis. In this reporting period, highlights from battery research include XAS studies of the oxidation phenomena in Li-ion battery cathode as materials undergo delithiation (J. Power Sources 418, 84–89 (2019)). Exploiting our *in-situ* capabilities allows new insights into the relationship between structure and functional properties of solar cells; including the effects of doping (ACS Nano 12, 7301-7311 (2018)), passivation (Nature 555, 497–501 (2018)) and annealing (Energy Environ. Sci. 11, 383-393 (2018)). Strategies to limit the non-radiative charge recombination in perovskite–polymer light-emitting diodes have also been explored (Nat. Photon. 12, 783-789 (2018)). Related studies on organic field effect transistors included the effects of embedding metallic nanoparticles (Appl. Phys. Lett. 113, 251903 (2018)) and the morphology of the bottom-contact organic layer (Langmuir 34, 6727-6736 (2018)) on their operational efficiency. In the nuclear industry, experiments have focused on hydrogen uptake in uranium metal-oxide bilayers which is critical in the safe storage of spent nuclear fuels (J. Nucl. Mater. 502, 9-19 (2018)). Partnerships continue to be nurtured between individual users and the Catalysis hub and industrial partners including Johnson Matthey, which benefit from our *in-situ* EXAFS and XAS capabilities. Recent highlights in catalysis research include selective studies of the catalytic oxidation of NH_3 to N_2 (Nat. Catal. 2, 157–163 (2019)) which exploited the capability of performing measurements at the Pd L-edges. Research has demonstrated how Pd nanoparticles stabilised in polymer immobilised ionic liquids and polystyrene are highly effective for the aqueous phase hydrogenation and sodium borohydride-based reductions, including the Suzuki-Miyaura cross-coupling reduction, for a wide range of nitroaromatic and heteroaromatic compounds under mild conditions (Catal. Sci. Technol. 8, 1454-1467 (2018) and Adv. Synth. Catal. 360, 3716–3731 (2018)).

Fundamental studies of magnetic materials continue on the beamline with the complex time dependence of curium metal explored through a combination of XMCD (on ID12) and diffraction on XMaS (Phys. Rev. B 99, 224419, (2019)). Resonant scattering at the Ru L-edge showed that the observed spin canting in $CeRu_2Al_{10}$ was localised on the Ce sites (J. Phys. Soc. Jpn. 87, 013706 (2018)) whilst the role of the orbital contribution to the magnetic moment of the transition-metal ion in isostructural weak ferromagnets was studied by conventional diffraction (Phys. Rev. B 98, 104424, 16 pp (2018)). Thin film structures continue to be studied with measurements allowing the thickness dependence of the Dzyaloshinskii-Moriya interaction in Co_2FeAl ultrathin films (Phys. Rev. Applied 9, 044044 (2018)) and the magnetostriction in nanolaminated Mn_2GaC MAX phases (Sci. Rep. 8, 2637 (2018)) to be determined.

Functional materials underpin a plethora of emerging technologies, with studies of these important materials continuing using combinations of XAS/WAXS/XRR and diffraction, in sample environments optimised for *in-operando* conditions. In soft matter systems the structures of self-assembled liquid crystals were studied (Adv. Funct. Mater. 28, 1804162, (2018) and Chem. Comm. 54, 156-159 (2018)). XRR was used to follow the structure of functional nanoparticles embedded in lipid bilayers (Nanoscale 10, 17965-17974 (2018)). Studies of 2D systems include the structure of graphene in aqueous media (Carbon 143, 97-105 (2019)) and a polar metal (Nat. Comm. 9, 1547 (2018)). Small atomic displacements were also measured by resonant diffraction in $SrTiO_3$ (Nat. Comm. 9, 178 (2018)).

In the area of **healthcare**, the *s*elf-assembly of fluoride-encapsulated polyhedral oligomeric silsesquioxane (POSS) nanocrystals was studied (Cryst. Eng. Comm. 21, 710-723 (2019)) along with detailed study of the crystallographic texture and mineral concentration in developing and mature human incisal enamel (Sci. Rep. 8, 14449 (2018)).
It is clear that output of XMaS impacts a wide range of scientific disciplines. The research activities are frequently singled out for inclusion in the ESRF's publications; 2018 ESRF Highlights Abdi-Jalebi M *et al. "Potassium passivation*

boosts luminescence and photovoltaic efficiency of halide perovskites" and Alsari M *et al.* "C*lean semiconductor behaviour of perovskite solar cells probed by simultaneous current-voltage and GIWAXS measurements"* as well as in the current 2018 ESRF Spotlights: Alsari M *et al. "Perovskite solar cells photovoltaic and structural evolution during film formation".* In addition, the beamline activities are disseminated in an annual newsletter and we are currently editing the 2019 version (available Feb. 2020) which will be circulated widely (>800 copies). Here we include a summary of the content of the current 2018 newsletter, which serves to showcase scientific case studies as well as the breadth of science undertaken along with the range of techniques available.

- **X-ray Emission Spectrometer at the XMaS Beamline** (Kvashnina *et al.*): *A description a new wavelength-dispersive configuration for enhanced energy resolution spectroscopy demonstrated on a CeO_2 sample.*
- **Picometer polar atomic displacements in $SrTiO_3$ determined by resonant x-ray diffraction** (Nat. Comm. 9, 178 (2018)): *A new approach of exploiting Resonant X-ray Diffraction (RXD) that makes use of strong fluctuations in the atomic scattering amplitudes to measure lattice parameters at the picometer level.*
- **Magnetostriction across the first order phase transition in Mn_2GaC MAX phase** (Sci. Rep. 8, 2637 (2018)): *A magnetostriction study of Mn_2GaC as it passes through an anisotropic magnetostructural transformation.*
- **Proximity-induced magnetism and Dzyaloshinskii-Moriya interaction at heavy metal/Co_2FeAl interfaces** (Phys. Rev. Applied 9, 044044 (2018)): *Resonant magnetic reflectivity exploring element specific interface induced magnetic proximity providing and allowing the interfacial Dzyaloshinskii-Moriya interaction (iDMI) to be studied.*
- **Towards a spatial and temporal model of human enamel biomineralization** (Sci. Rep. 8, 14449 (2018)): *An XRD study of human dental enamel at different stages of tooth development.*
- **Trigonal columnar self-assembly of bent phasmid mesogens** (Chem. Comm. 54, 156-159 (2018)): *Exploring trigonal non-cylindrical columnar liquid crystal phases with an unusual three-fold rather than six-fold symmetry.*
- **Surface structure of few layer graphene** (Carbon 143, 97-105 (2019)): *An x-ray reflectivity study of commercial single layer graphene sheets as a function of temperature.*
- **Clean semiconductor behaviour of perovskite solar cells probed by simultaneous current-voltage and GI-WAXS measurements** (Energy Environ. Sci. 11, 383-393 (2018)): *By exploiting interdigitated back-contact (IBC) solar cells, simultaneous opto-electrical and GI-WAXS measurements were measured during in situ annealing.*
- **New tool for simulation of surface resonant x-ray diffraction** (J. Chem. Theory Comput. 14, 973-980 (2018)): U*sing new ab-initio DFT approaches, modelling paradigms for fitting surface resonant XRD spectra are demonstrated.*
- **Probing the hydriding behaviour of a buried uranium interface with synchrotron radiation** (J. Nucl. Mater. 502, 9 (2018)): *In situ temperature dependent XRD revealing how hydrogen is incorporated into uranium oxides.*
- **Using NEXAFS, XPS and TDDFT to probe the electronic structure of ionic liquids** (Faraday Discuss. 206, 183-201 (2018)): *S K-edge NEXAFS study of sulphur-containing ionic liquids which when modelled by DFT show that excited states are not always localised on the ion being probed using S 1s NEXAFS spectroscopy.*

A full and up-to-date publication list is maintained on the XMaS webpages with output covering this period including:

Al-Mosawi M, *et al.* Sci. Rep. 8, 14449 (2018)
Bikondoa O, *et al.* Phil. Trans. R. Soc. A 377, 20180237 (2019)
Blidberg A, *et al.* J. Power Sources 418, 84 (2019)
Chapman JBJ, *et al.* J. Am. Ceram. Soc. 101, 874 (2018)
Dann EK, *et al.* Nat. Catal. 2, 157 (2019)
Heeley E, *et al.* Cryst. Eng. Comm. 21, 710 (2019)
Huss-Hansen MK, *et al.* Appl. Phys. Lett. 113, 251903 (2018)
Hussain H, *et al.* J. Phys. Chem. C 123, 13545–13550 (2019)
Gründer Y, *et al.* Surf. Sci. 680, 113 (2019)
Lander GH, *et al.* Phys. Rev. B 99, 224419, (2019)
Lehmann A, *et al.* Chem. Comm. 54, 12306 (2018)
Lehmann A, *et al.* Adv. Funct. Mater. 28, 1804162, (2018)
Pincini D, *et al.* Phys. Rev. B 98, 104424, (2018)
Sirovica S, *et al.* Macromolecules 52 (14) 5377 (2019)
Stevenson W, *et al.* Phys. Chem. Chem. Phys. 20, 25268 (2018)
Thompson P, *et al.* AIP Conf. Proc. 2054, 060030, (2019)
Tripathy A, *et al.* Colloids Interfaces 2, 74 (2018)
Treacy J.P.W, *et al.* J. Phys. Chem. C 123, 8463 (2019)
Torrelles X, *et al.* Acta Cryst. B 75, 830 (2019)
Wlodek M, *et al.* Nanoscale 10, 17965 (2018)
Zhao B, *et al.* Nature Photonics 12, 783 (2018)
Zhou L, *et al.* Carbon 136, 255 (2018)
Zhou L, *et al.* Carbon 143, 97 (2019)

Publications for the general public:

A 32 min video with Prof. Sir Richard Friend *et al.* (University of Cambridge) was produced by Scientific Video Protocols (SciVPro) (doi.org/10.32386/scivpro.000005). Entitled "All you need to know about back-contact photovoltaics", the data discussed during Dr Abdi-Jalebi and Alsari's interview were collected on XMaS. The video had 3490 views since its publication on 12th March 2019. The facility was also represented as part of the *UNESCO 2018 International Year of Light* (Lightsources.org organised a PhotoWall) and *World Environment Day* (twitter).

Impact: Training, Outreach and Societal Impacts (max. 1 page):

Activities to promote the facility beyond its core user base:

As well as hosting an annual user meeting, producing an annual newsletter and having a presence on the web and social media (@XMaSBeam), we also sponsor UK meetings to promote our activities and build new collaborations. On Feb 4-5, 2019 XMaS sponsored the X-ray and Neutron Scattering in Multiferroic and Ferroelectric Materials Workshop (5th meeting) which took place at the IOM3 in London. This meeting brought together international experts from the multiferroics, magnetoelectrics and ferroelectrics communities with neutron, synchrotron and other spectroscopy facility users to present the latest developments in this field. We encourage exploratory studies from new academic and industrial user communities to be instigated through in-house research collaborations and are open to designing, building and commissioning new sample environments. Input from the User community was instrumental in formulating the refurbishment and upgrade programme that the beamline is currently undergoing to prepare for operations in the new ESRF lattice in August 2020. The capabilities of XMaS are presented at focused meetings, such as at DIAMOND and other SR facilities, where we engage with the wider SR community. Direct engagement with industry takes place through "Industry days" at the University of Warwick and with DISCO (the Diamond industrial user committee).

Public engagement:

In July 2019, for the fourth year running, we held the XMaS Scientist Experience, an outreach activity that tackles gender bias in Physics. Again, 16 winners from a nationwide competition targeted at Year 12 female Physics students were taken to the EPN campus where they participated in the Synchrotron@School programme (Figure 7) and interacted with a number of female scientists. The aim of the XMaS Scientist experience is to encourage young women to consider careers in STEM by showing them possible job opportunities they would have been unaware of and introducing them to inspirational role models in an international setting. The trip received a lot of attention on social media (@XMaSSchoolTrip, @XMaSBeam) and was publicised on BBC Radio 2 Coventry and Warwickshire, with a 2018 participant and current University of Warwick Physics student and one of the 2019 participants taking part in the Vic Minnett Show on 17th July. A podcast is available from the BBC webpage.

Figure 7: Competition winners taking part in the Synchrotron@School programme.

The winners returned to the University of Warwick in September for our yearly XMaS Scientist Experience Student Showcase. Feedback from both winners and their parents was very positive. Maggie Nichols stated that "*The experience being only for girls allowed me to be more confident and comfortable sharing ideas and working as a group.*" Orlagh Ward found that "*Meeting the scientists for lunch was such a good idea, as it allowed for us all to see how they have come through life to that point, to maybe encourage us to do the same.*"

Competition entrants come now from schools located as far as Kent and Essex, indicating the growing national reach of the activity as, historically, entrants came only from schools located in the Liverpool and Coventry areas, as well as adjacent counties. We have now started collaborating with the Warwick Scholars team to reach more students from Widening Participation backgrounds, with the possibility of devoting specific spaces on the next Scientist Experience to females from such backgrounds, thus broadening the outreach agenda.

Science Gala:

XMaS also sponsors and organises a Science Gala evening for school pupils of all ages at Warwick, with the fifth edition taking place on 30th January 2019. This event is the launch event for the XMaS Science Experience and will incorporate the Synchrotron@School programme in 2020. For the third year running, the evening attracted around 750 members of the public, who were able to tour laboratories, take part in experiments and workshops, attend lectures and discuss careers in STEM with scientists. We had over 40 exhibitors, with an increased focus on inter-disciplinarity. The event was well publicised in the media (the Coventry Telegraph, BBC Radio Coventry and Warwickshire (Vic Minnett Show, 29/01/19, Twitter:@gala_uo). Feedback from both the public and exhibitors was extremely positive.

Impact: Economic Impact (max. 1 page): Please provide evidence of the economic impact that the facility has had.

Examples of where work supported by the facility has led to wealth creation and/or attracted inward investment: It is difficult to track directly the economic impact of the research undertaken on the facility as this impact is often several stages removed from the fundamental scientific studies that are performed. We actively ask our users for details of industrial collaborations associated with beamtime and this is logged in our database. The interactions are with central facilities as well as national and international companies including, for example, ABB, Siemens, BP, the Nuclear Decommissioning Authority, US Navy, Electrosciences Ltd and the Surrey 5G centre. Direct inward investment has been through proprietary beamtime purchased through the ESRF by Johnson Matthey.

Information on projects which have led to the development or improvement of products and procedures: The beamline staff are involved with ISO/TC 201 (Surface Chemical Analysis). We are experts on the BIS committee CII/60 which mirrors TC 201 and are working with international partners to define new ISOs and hence British standards on the analysis of x-ray reflectometry (XRR) and x-ray fluorescence (XRF) spectroscopy. Within an EU framework this is being supported by the newly funded COST Action "ENFORCE - European Network for Chemical Elemental Analysis by Total Reflection X-Ray Fluorescence" for which we are workgroup leaders in standardisation. The activities of both groups include developing new standards and round-robin tests using SR and lab-based sources with the aim of providing the average analyst the metrology tools for traceable and quantitative analysis.

Information on collaborative projects with industry that have been supported by the facility: XMaS has continually evolved to meet a broad range of scientific challenges and this has required continual development of both sample environments and beam conditioning components. For example, a new optical microscope to observe x-ray excited light from materials has been developed to study the spatial variations in materials without the need for nano-focused beams with high power density (Heritage Science 3:14 (2015). To date XMaS holds 9 licence agreements with 3 different companies which have resulted in over £1M of sales.

Related publications that have industrial or international co-authorship [2012-2019]: Consistently 70% of the publications have international co-authorship, and 45% are co-authored with collaborators at central facilities. Such close collaboration with other central facilities ensures best practice is quickly shared and implemented between service providers. In addition, papers within the healthcare field have potential impact in therapeutics (Bioelectrochemistry 110 41-45 (2016) , Micron 83 48 (2016)), restorative dental treatments (Mater. Today 17 7 312 (2014)) and new cheaper detectors of medical imaging (Nat. Photon. 9 842-848 (2015)).

Information on any additional funding or leverage that has been received: Within the framework of the *EMRP Nanostrain Project* we leveraged additional funding for a 2-year PDRA position **£194k**. The success of the *Nanostrain Project* resulted in a successful submission of a follow-up EMPIR JRP programme "*Metrology for advanced energy-saving technology in next-generation electronics applications* - ***ADVENT***" to a value of **€94,688** for XMaS. The beamline team has also recently applied for two more EMRP funded programmes. The first one, "*JRP g-10 OpMetEnergy - Operando Metrology for Energy Storage Materials*", will develop traceable measurement tools and methods for *operando* characterisation of energy storage materials under dynamic charge/discharge conditions, establish best practice for implementing *operando* techniques to inform new materials development and build a metrological framework for linking these *operando* measurements to traceable, *ex situ* structural and chemical analysis. As part of this consortium, XMaS hopes to receive **€100,000**. The second, "*JRP g-19 Ageing and Fatigue of Energy Materials*", will develop new metrology techniques for traceable measurement of ageing-related phenomena in energy materials. This will be fulfilled by developing methods that probe those parameters that are affected by ageing and by bridging the gap between in-field techniques and nano/microscale analytical methods. If this project is accepted, XMaS hopes to receive funding to the value of **€125,000**. A further **£18k** was received to support our outreach activity from the University of Warwick Widening Participation fund as well as **~£5k** per annum from the Science faculty at Warwick. The facility also supports CDT training programmes, providing bespoke training courses in Grenoble on request. XMaS is also part of the materials characterisation suite at the University of Liverpool following a successful equipment bid to EPSRC (EP/P001513/1) for x-ray diffraction capabilities.

Details of any other activities that have involved industrial participation: Large collaborative projects that we are developing are closely aligned with energy materials and our partners include the 5G centre at the University of Surrey, IBM Zürich, the US Naval labs and Electrosciences Ltd. We are also working on expanding our green chemistry and catalysis research programmes in partnership with British Petroleum, the catalysis Hub at RAL and Johnson Matthey.

International Interactions (1/2 page):

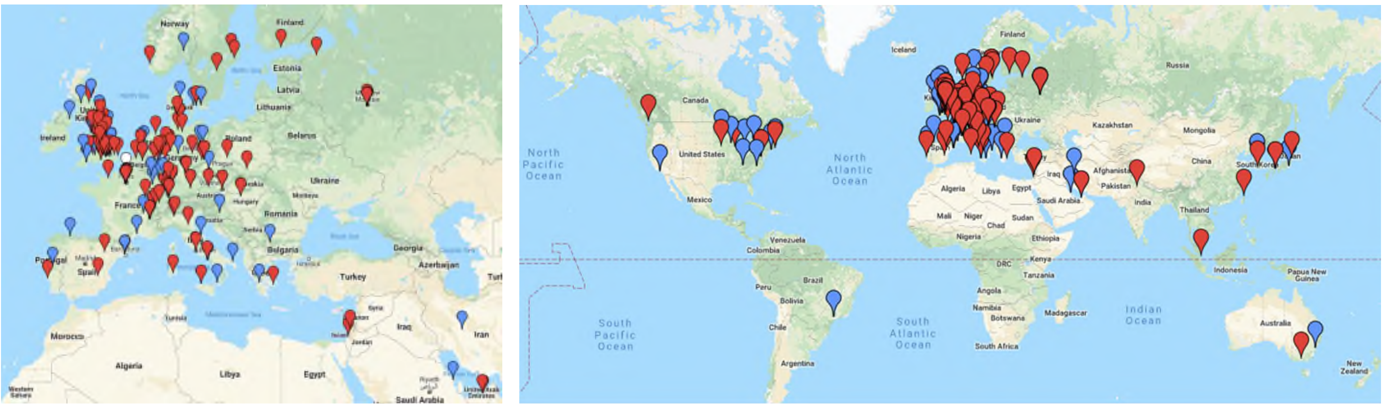


Figure 8: Reach of the XMaS facility since it began supporting UK materials researchers and their collaborators. Red markers correspond to users of the facility since 2012.

XMaS provides the UK science base with direct access to the EPN campus, placing EPSRC-funded science at the forefront of European research. Access to EPN support facilities (including the Partnership for Soft Condensed Matter) is guaranteed through our ESRF contribution and comes with no additional cost. Synchrotron studies tend to be international in nature and the facility is well placed to facilitate and foster strong international collaborations, greatly enhancing the research, impact and training quality for the UK community. Our user community and its collaborators extend around the world (Figure 8). Since 2012, ~70% of papers have international co-authorship. XMaS has a strategic partnership with sectors 4 and 6 of the Advanced Photon Source (APS) in the US in the development of new x-ray metrologies and sample environments. Over the current reporting period, which maps into the ESRF shutdown (Dec. 2018 – Aug. 2020), XMaS users have been able to apply for beamtime at the APS (sectors 4, 6) under a special agreement. This arrangement will be reciprocated for APS users during the APS upgrade shutdown. We are actively involved in the European Synchrotron Users Organisation and are looking to host several PhD students through CDT and Marie-Curie networks.

Improvement (max. 1 page): Please indicate steps that have been taken to improve the access, user experience and ensure the long-term sustainability of the facility.

From December 2018 until the summer of 2020 the ESRF is installing and commissioning its new magnetic lattice; which also includes a new source for the XMaS beamline. Following detailed discussions with the user community, and ensuring their evolving needs were met, a 0.86 T short-bend magnet was selected as the new source. Coupled with the new storage ring, this will significantly increase the capability of the beamline; enabling operations to higher x-ray energies (from a cut-off of 15 keV to ~40 keV) coupled with a much smaller and brighter beam. As the source point of the beamline has moved, we have redesigned the entire beam delivery system, including new optical elements and upgrades to essential safety features. A complete refurbishment and updating of the key beamline components is being performed. The following is a list of actions taken in this direction:

- HUBER Diffraktionstechnik GmbH & Co. is completely refurbishing the (>20-year-old) diffractometer. An additional strengthened 2-theta arm is being machined and will provide two, offset, detection pathways so that data can be collected using either 2D or point detectors. By installing computer-controlled slides, changing configurations will be more streamlined and increase operational efficiency.
- We are replacing the old water-cooled monochromator by a liquid nitrogen cooled version, which we have developed with new high-side load actuators. The new system is capable of operating down to 2.1 keV.
- We are replacing the 20-year-old toroidal mirror in the optics hutch by a new system with two interchangeable mirrors coated with Cr and Pt stripes, respectively. This will allow continuous tuning of the x-ray energy from 2.1 to 40 keV. Reduced aberrations in the mirror finish also results in a smaller, brighter beam being delivered to the sample.
- We are updating the beam delivery system to allow smoother and more efficient changes between experiments and to ensure that the incident beam interacts with the same sample volume across the entire energy range (Figure 9). Significant enhancements have been developed to make spectroscopic studies more quantitative and efficient.
- We are purchasing a new suite of 2D pixelated X-ray detectors: a CdTe detector to work at high energies up to 40 keV and a relatively fast large area detector to replace our obsolete MAR-CCD camera. Coupled with software updates, the detectors will improve data collection efficiency and allow scattering space to be probed in three dimensions.
- We are replacing the old VME motor controllers with IcePAPs, the latest ESRF standard, and a complete overhaul of the SPEC control environment is underway. This improvement ensures uptime and makes configuration changes more efficient.
- New data and analysis pathways are under development to ensure that users can interpret data more rapidly and allow real-time decisions to be made on experimental methodologies.

We have designed the beam delivery system to ensure that our current capability of providing an unfocussed monochromatic beam and white beam operational modes (both in addition to the normal monochromatic beam) is retained after the EBS upgrade.

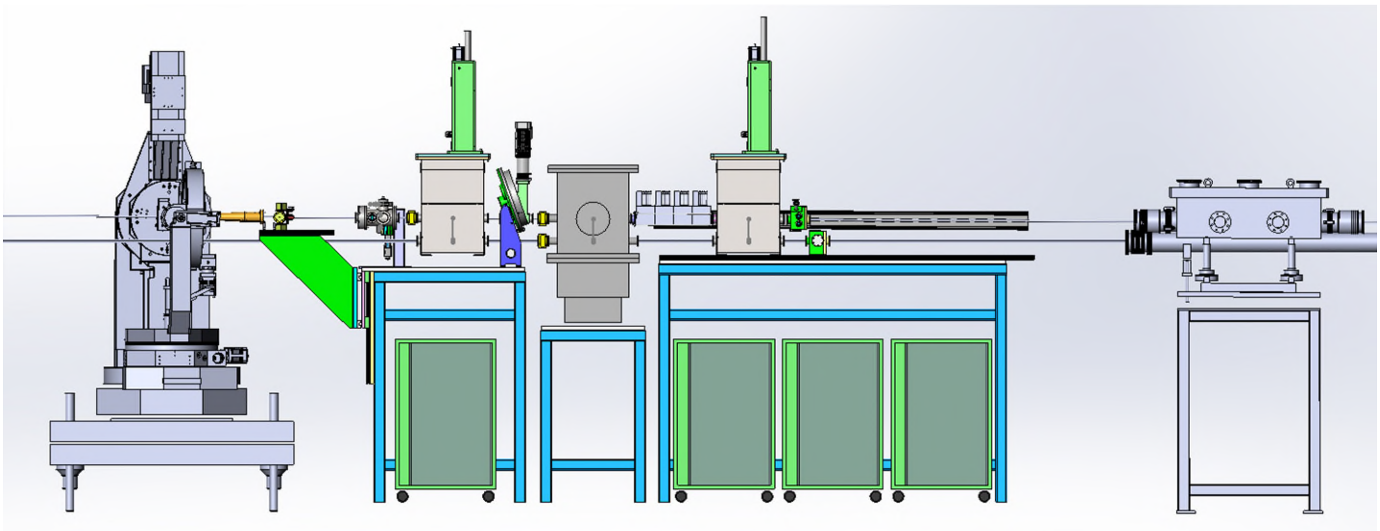


Figure 9: Sketch of the new delivery system inside the experimental hutch. The beam enters from the right.

Strategic Fit (max. 1/2 page): Please provide evidence on how the facility fits the strategic priorities of EPSRC and state any actions taken / will be taken to align the facility with these strategic priorities.

Much of the scientific programme at the XMaS National Research Facility is contained within the Physical Sciences remit of EPSRC. As noted in the recent infrastructure roadmap, advances in materials characterisation "increasingly relies on sophisticated experiments at a range of bespoke national and international facilities, often at the leading edge of what is technically possible". Within this context XMaS provides the UK community with the necessary tools for transformative materials development. The current UK instrumentation portfolio has been developed over the last 20 years, with XMaS recognised as having an important role that underpins the wider materials science communities across the UK and their international collaborators. The flexibility of the beamline design coupled with technological developments has enabled an ever-broadening range of scientific challenges to be addressed. The demonstrable ability to adapt to the changing scientific challenges and landscape shows that the facility remains flexible to the strategic requirements of EPSRC and other UK stake holders.

The principal aim of the XMaS facility is to support world-leading research, discovery and innovation by providing a set of unique materials research capabilities to the UK science base. We provide creativity within the science community to stimulate innovative solutions through advanced materials research driving new processes, products and sustainable solutions. The facility is a sustainable asset to support design, build and test the next generation of transformative technologies with characterisation tools across a range of relevant length and time scales. Our upgrade plans facilitate and support the development of new metrologies, as well as providing a proving ground for advanced materials with novel chemical, physical or mechanical properties. Thus, the balance of science that is performed at XMaS encompasses both long-term discovery-led research and shorter-term impact cases that contribute to the development of a productive, connected, resilient and healthy nation.

The combination of a state-of-the-art ESRF storage ring and photon source, high quality technical and scientific support and a strong international user community has maintained the beamline at the leading edge of materials characterisation. The continual development of novel sample environments enables XMaS to maintain a diverse scientific programme cutting across the challenge themes which complements and strengthens UK-based facilities. As materials discovery continues apace it is evident that the XMaS beamline has an important strategic role in providing fundamental and technologically relevant atomic-scale information to researchers in the physical and life sciences. Due to the location of the XMaS facility at the ESRF, which itself is part of the larger EPN campus, there is a natural environment for UK researchers to adopt an international approach and form collaborative projects. In a possible post-Brexit research environment, it is essential that UK scientists are able to maintain, develop and nurture links with international colleagues and facilities. XMaS will play a key role in maintaining engagement with EU collaborators providing an access point for UK users to experience the ESRF and develop collaborative projects both at the ESRF and ILL that go beyond the capabilities of the XMaS beamline.

Sustainability (max ½ page): Please provide an update on the facility plans towards greater sustainability and efficiency.

As seen in Figure 2, the facility has made significant efficiency gains over the past five years, increasing throughput and maximising the number of user visits. This has been accomplished within the same operational budget. The majority of the changes currently being implemented on the facility are focused on improving efficiency. Our upgrade ethos has been to minimise down time as the facility is reconfigured for different experiments. The new monochromator and mirror system will allow fixed-exit height operations allowing delivery systems to be homed and zeroed rapidly. These upgrades, coupled with the increased brightness, are expected to further increase throughput by 10-20% over the coming years.

In terms of data analysis pipelines, the facility is reaching out to DIAMOND to harmonise resource and share in developments so that users can move more seamlessly between facilities. This approach is to be supported by two new PDRAs on the beamline whose role will be to work with users and help identify data visualization, minimisation and fitting strategies. Such an approach will be essential in the future as DIAMOND undergoes its own upgrade (shutdown between 2025 and 2027) when XMaS will be even more important to the UK materials community. To this end, we have also implemented new approaches to engage with industrial users. Operational days are now reserved for industrial access, either directly or for developing industrial case studies.

To engage with new users, we are currently developing a suite of case studies to highlight our capabilities and give potential new users detailed information on the experimental techniques that are available. The increasing focus on more efficient data collection and data analysis pathways will ensure that inexperienced users can derive the same impact as our long-term users. In this way the user community will continue to grow, be flexible to the changing needs of the materials communities and remain at the forefront of advance materials characterisation using synchrotron x-rays.

Summary (max ½ page): Please provide a short summary of the highlights from the annual report for this reporting year

This has been a most unusual year for XMaS operations because it has overlapped very significantly with the ESRF's installation of its new magnet lattice (the Extremely Brilliant Source – EBS project) which, of course, necessitated the cessation of the XMaS User programme for the time being. With the accompanying essential commissioning work there were no user experiments in the period of this particular annual report and that is why we have chosen to report on a wider period to convey the depth and range of typical beamline activity. As detailed in the report above, new users continue to be attracted to the facility and experimental results continue the trend of being increasingly published in high impact journals and highlighted by the ESRF.

ESRF's new EBS provides XMaS with higher brightness (typically an order of magnitude) and extends the upper energy range from 15 keV to over 30 keV whilst retaining the current low energy capability down to 2.1 keV. However, the new magnet lattice came with the challenge of major adaptations to the beamline; namely it had to be moved "sideways" by more than 100 mm and lengthened by several metres to preserve the 1:1 beam focussing. The latter necessitated a major extension of the lead-walled experimental hutch and the construction of a new control cabin which had to be completed during the late summer/autumn of 2018. Most significantly the hiatus in user operation has provided us with the much-needed opportunity to upgrade the beamline after 21 years of operation and take advantage of the new higher energy range and the overall higher brightness, with the aim of attracting new users and being able to operate faster experimental turnaround. Thus, we have installed a new cryo-cooled monochromator, soon to be accompanied by the installation of focussing mirrors covering the extended energy range and a completely refurbished diffractometer – arguably the beamline's centrepiece facility. New diagnostic and beam conditioning optics will further ensure that the capabilities of the refurbished beamline are enhanced and we actively prepare for a scheduled return to user operations in the late summer of 2020.

Case Study 1

1. Title of Case Study: Clean semiconductor behaviour of perovskite solar cells probed by simultaneous current-voltage and GIWAXS measurements

2. Grant Reference Number: XMaS

3. One sentence summary: Employing simultaneous *in situ* grazing incidence wide angle x-ray scattering (GIWAXS) and *operando* current-voltage measurements on metal-halide perovskite interdigitated-back-contact (IBC) solar cells, the remarkably clean semiconductor behaviour of perovskites films is shown to emerge in the earliest phase of conversion from the as-coated precursor film.

4. One paragraph summary: Metal-halide perovskites show remarkably clean semiconductor behaviour, as evidenced by their excellent solar cell performance, in spite of the presence of many structural and chemical defects. This study shows how the clean semiconductor performance sets in during the earliest phase of conversion from the metal salts and organic-based precursors and solvent, using simultaneous *in situ* synchrotron x-ray scattering and *in operando* current-voltage measurements on films prepared on interdigitated back-contact substrates. At the first stages of conversion from the precursor phase, at the percolation threshold for bulk conductance, high photo-voltages are observed, even though the bulk of the material is still present as precursors. This indicates that at the earliest stages of perovskite structure formation, the semiconductor gap is already well-defined and free of sub-gap trap states. The short-circuit current, in contrast, continues to grow until the perovskite phase is fully formed, when there are bulk pathways for charge diffusion and collection. This work reveals important relationships between the precursors conversion and device performance and highlights the remarkable defect tolerance of perovskite materials.

5. Key outputs in bullet points:

- Photovoltaic device performance improvement
- Simultaneous *in operando* current-voltage and *in situ* x-ray scattering measurements
- PhD of M. Alsari
- Collaboration between XMaS and Dr. Samuele Lilliu

6. Main body text:

Perovskite photovoltaics (PV) is one of the fastest growing opto-electronic technologies with device efficiencies currently exceeding 23% in single solar cells and 27.3% in tandem devices [1]. To further improve device performance, researchers are exploring a variety of optimisation strategies, such as thermal engineering. For most solution-processed perovskite materials, annealing temperature and duration are critical factors for optimised conversion of the as-coated precursor material into a functional polycrystalline perovskite film. In this work, Alsari and co-workers [2] employ a new investigation method illustrated in **Fig. 1** that can significantly reduce the workload required in thermal engineering of perovskite solar cells and, at the same time, establish a direct correlation between their opto-electrical and structural properties during *in situ* annealing. This is achieved by exploiting the concept of interdigitated back-contact (IBC) solar cells. Here the electron (TiO_2) and hole (PEDOT) selective electrodes are co-positioned on the backside of the cell in an interdigitated fashion (**Fig. 1b**)[3].

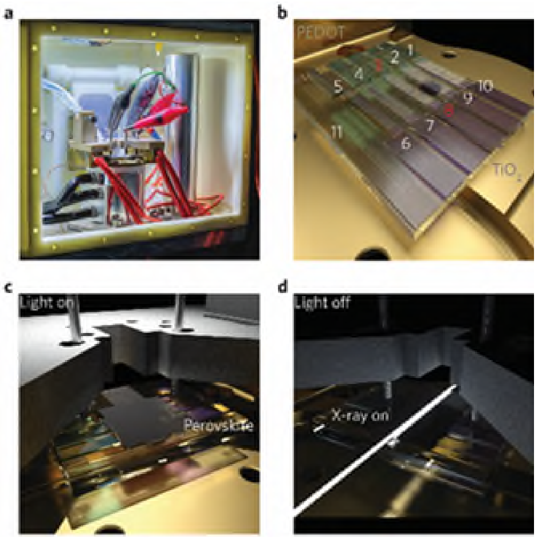


Figure 1: Illustration of the measurements setup: a) annealing chamber; b) ITO integrated substrate electrodeposited with PEDOT (electrode 1-5) and TiO_2 (electrodes 6-10); c) the experimental setup for current-voltage sweeps performed in situ without x-rays; d) the setup for diffraction pattern measurements (light off) using grazing incidence geometry.

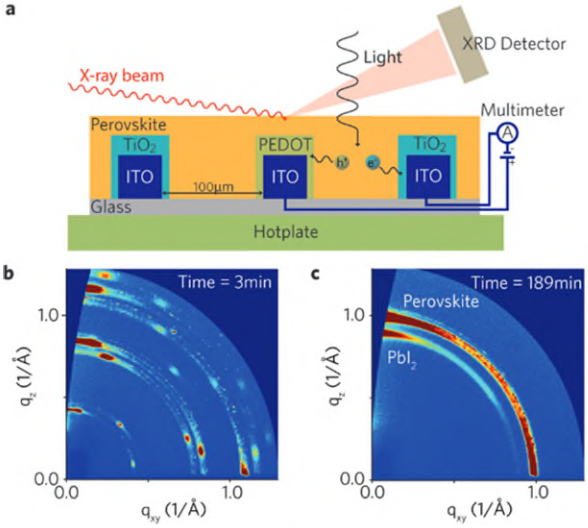


Figure 2: Simultaneous GIWAXS and current-voltage measurements of an IBC solar-cell during in situ annealing at 88.2°C . a) Experimental geometry showing the grazing incident x-ray beam impinging onto the sample and the detector set to collect WAXS pattern. The electrodes connected to the current source and voltmeter are also shown; b) diffraction pattern after about 3 min of annealing showing only the precursor phase; c) diffraction pattern after about 3 h of annealing showing the perovskite and the lead iodide phases.

The main advantage of IBC solar cells is that the perovskite layer represents the final step of the device fabrication. Because the perovskite film is unobstructed by any top layer, *in situ* annealing can be performed without compromising the film formation. At the same time, the perovskite film is directly accessible by an x-ray beam, making IBC devices a great solution for simultaneous opto-electrical and grazing incidence wide angle x-ray scattering (GIWAXS) measurements performed *in situ* during annealing (**Fig. 2**). With the setup developed at the XMaS beamline, Alsari *et al.* demonstrate a high throughput thermal engineering route that can be used on a variety of perovskite materials, along with the possibility of establishing a direct correlation between the figures-of-merit of a solar cell and its structural properties. **Fig. 3** shows a summary of the photovoltaic and structural measurements on $CH_3NH_3PbI_3$ perovskite IBC solar cells. The remarkably clean semiconductor behaviour of perovskites is evidenced by the high photo-voltages measured at the first stages of perovskite conversion from precursors, at the percolation threshold for bulk conductance. The open circuit voltage (V_{oc}) reaches a maximum value before the precursor has fully converted into perovskite, when the fraction of precursor and perovskite crystals is comparable (**Fig. 3 c-d**). The short circuit current (J_{sc}) and power conversion efficiency (PCE) follow a trend similar to that of the perovskite peak intensity extracted from the GIWAXS measurements. The measurement strategy followed by Alsari *et al.* is not limited to perovskite materials and is of interest for any solution processable photovoltaic technology requiring thermal annealing.

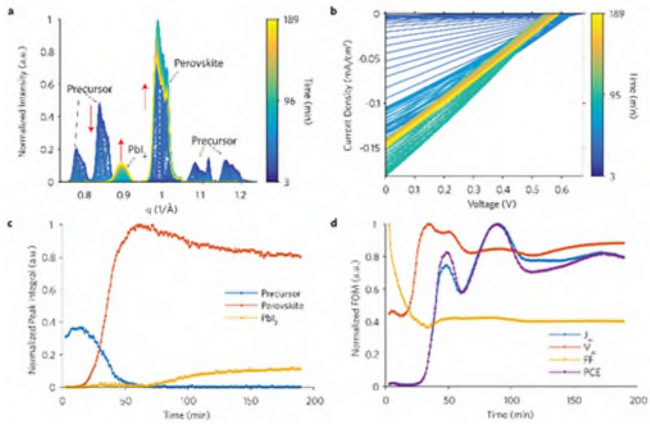


Figure 3: Structural and opto-electrical parameters extracted from simultaneous GIWAXS diffraction patterns (under dark conditions) and current-voltage measurement (under light conditions) of a perovskite ($CH_3NH_3PbI_3$) IBC solar cell during in situ annealing [1].

[1] H.J. Snaith and S. Lilliu, SciVPro (2018) 1, 1. doi:10.32386/scivpro.000004
[2] M. Alsari *et al.*, Energy Environ. Sci. (2018) 11, 383.
[3] L.M. Pazos-Outón *et al.*, Science (2016) 351, 1430.

7. Names of key academics and any collaborators:

Dr. Samuele Lilliu, Department of Physics and Astronomy, University of Sheffield

8. Sources of significant sponsorship (if applicable):

XMaS
The President of the UAE's Distinguished Student Scholarship Program (DSS), granted by the Ministry of Presidential Affairs, UAE
University of Sheffield DTG account
CDT in New and Sustainable PV, EP/L01551X/1

9. Who should we contact for more information?

Dr. Samuele Lilliu, Department of Physics and Astronomy, University of Sheffield, s.lilliu@sheffield.ac.uk, +44 (0)114 222 3501

Case Study 2

1. Title of Case Study: A spatial and temporal model of human enamel biomineralisation

2. Grant Reference Number: XMaS

3. One sentence summary: Fundamental understanding of spatial and temporal progression of matrix-mediated dental enamel biomineralisation using synchrotron x-ray diffraction is key to developing biomimetic approaches to repair and replace lost enamel tissue.

4. One paragraph summary: Human dental enamel biomineralisation is a phenomenon whose timing and spatial progression is poorly understood. For dental and skeletal tissue research, understanding organic matrix-mediated mineralisation holds the key to developing successful reparative or regenerative hard tissue biomimetic medical and dental technologies. Our aim is to use synchrotron x-ray diffraction (SXRD) on human teeth at different stages of development to provide novel insights into the biomineralisation process in human dental enamel to inform emerging technologies in regenerative and reparative dentistry.

5. Key outputs in bullet points:

- Timing and spatial progression of human dental enamel biomineralisation
- Information for emerging technologies in regenerative and reparative dentistry, including the work leading to publication [1] and European patent [2]
- PhD of Mohammed Al-Mosawi at QMUL (2018) leading to PDRA at University of Leeds (2019)
- Collaboration between XMaS and Prof. Maisoon Al-Jawad

6. Main body text:

Understanding the temporal and spatial progression of human dental enamel biomineralisation holds the key to developing successful reparative or regenerative hard tissue biomimetic medical and dental technologies. In this study, five human central incisors at different stages of enamel maturation were spatially mapped using synchrotron x-ray diffraction and x-ray microtomography techniques. Our aim was to provide novel insights into the biomineralisation process in human dental enamel to inform emerging technologies in regenerative and reparative dentistry.

Synchrotron x-ray diffraction (SXRD) using XMaS at the ESRF and B16 at DLS, combined with x-ray absorption microtomography (XMT) and quantitative backscattered electron (qBSE) imaging were used to generate crystallographic orientation, mineral density, and micro-morphology maps of the five human central incisors at different stages of enamel maturation taken from an archaeological collection. Experimental details can be found in the published article [3].

XMT revealed a bi-directional mineralisation "front" starting vertically at the dentine horn tip and horizontally at the enamel-dentine junction (EDJ) travelling cervically and peripherally as a function of enamel maturation until the relative mineral density was uniform in the fully mature tooth at 2.75(1) g.cm^{-3} (not shown here but available in [1]). Analysing azimuthally, 2D SXRD patterns revealed that within any probed region, two co-localised groups of crystallites ("populations") existe, distinguishable by their different orientation and alignment properties. One population of crystallites contributed a higher percentage, by a factor of ~4, to the overall crystalline structure (**Fig. 1a**). The angular separation between the populations was ~40°, varying as a function of position within the tooth crown (**Fig. 1b**). Interestingly, the population displaying a considerably lower degree of crystallite alignment (**Fig. 2a** – higher full width at half maximum (FWHM) values compared to **Fig. 2b**) contributed the higher percentage to the overall crystalline structure. These phenomena were observed in all stages of tooth development with the precise spatial distributions varying somewhat at each developmental stage. qBSE analyses suggested that the two observed populations are most likely due to spatial changes in crystallite bundle (prism) orientations (known as prism decussation), and revealed that mineralisation of prism cores precedes that of prism boundaries (more information available in [3]).

Quantification of the direction and magnitude of orientation and alignment within two distinct populations of crystallites in developing and mature enamel has not been shown previously. These results allow us to understand the development of the complex hierarchical enamel structure and provide new insights towards a quantitative spatio-temporal model of human enamel biomineralisation.

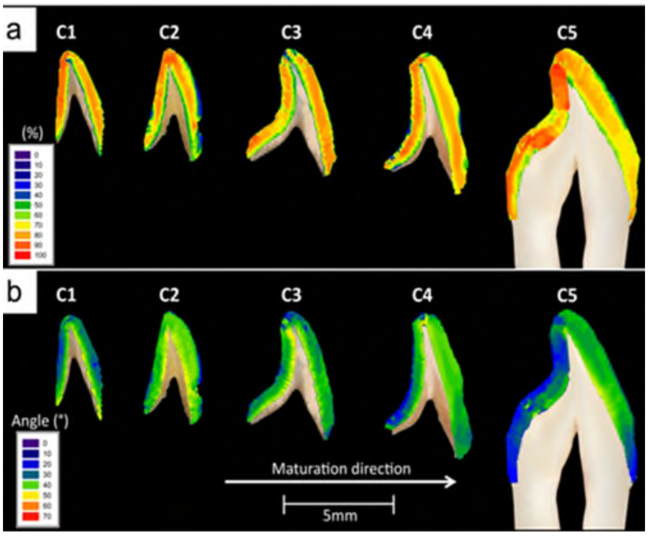


Figure 2: (a) Percentage of crystallites belonging to the first orientation population and (b) the angle between the two orientation populations.

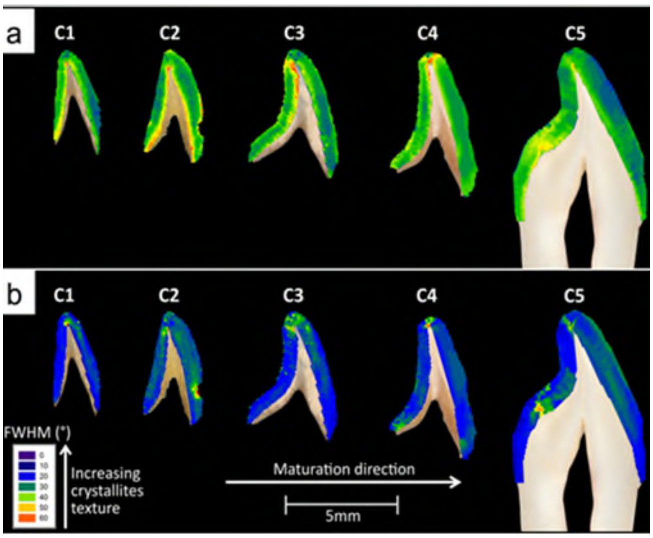


Figure 3: Texture magnitude distribution of crystallites of (a) first and (b) second orientation populations.

[1] Elsharkawy S, Al-Jawad M *et al.*, Nature Communications (2018) 9, 2145

[2] Elsharkawy S, Al-Jawad M, Chavarria AM, Tejeda-Montes E, Sanchez RSR. (2019) EP3436474

[3] Al-Mosawi M, Davis GR, Bushby A, Montgomery J, Beaumont J & Al-Jawad M, Scientific Reports (2018) 8, 14449

7. Names of key academics and any collaborators:

Prof. Maisoon Al-Jawad (School of Dentistry, Faculty of Medicine and Health, University of Leeds)
Prof. Janet Montgomery (Department of Archaeology, Durham University)
Dr. Julia Beaumont (School of Archaeological and Forensic Sciences, University of Bradford)

8. Sources of significant sponsorship (if applicable):

XMaS
NERC grant “Timelines in Teeth” NE/F019096/2 (previously EPSRC EP/D066298/1)

9. Who should we contact for more information?

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Assessment Criteria:

The assessment panel will assess the Annual Report against the criteria outlined below, scoring each with a score from 1-6. At the end, an overall score will be given. The score and feedback should enable the facility, together with their steering committee and EPSRC contact to ensure the best possible service is provided to the user community.

1) **Key Performance Indicators (KPIs) and Service Levels (SLs):** Over the reporting period, did the facility meet all KPIs and SLs laid out in the contract (score 6 = all KPIs and SLs met, very high standard / score 5 = all KPIs and SLs met, high standard). If not, did the facility do their best in mitigating negative impact for the users and did the facility take steps to improve performance in the future? To which degree where these steps successful? (not very successful: score 2, partially successful: score 3, predominantly successful: score 4). If the facility has not met more than 2 of their KPIs and SLs AND has not taken any steps to mitigate effects and improve performance, score 1).

2) **Scientific Excellence and Users:** Does the facility support scientific excellence in the UK to its user community? Does the facility actively engage with a variety of user communities and provide support for special needs (students, business collaborations etc.)? (1: Does not meet the criteria in any way; 2: partially meets the criteria, but with major weaknesses; 3: partially meets the criteria, but there is room for improvement; 4: meets the criteria; 5: meets the criteria and sometimes exceeds expectations; 6: exceeds expectations)

3) **Impact: Training, Outreach, Societal and Economic Impact:** In what way does the facility support the generation of broader impact, such as training of skilled people, enabling broader societal and/or economic benefits of scientific work e.g. through collaborations, and promote the Engineering and Physical Sciences among the wider public. (1: Does not meet the criteria in any way; 2: partially meets the criteria, but with major weaknesses; 3: partially meets the criteria, but there is room for improvement; 4: meets the criteria; 5: meets the criteria and sometimes exceeds expectations; 6: exceeds expectations)

4) **Improvement**: Has the facility thought of ways to improve the user experience and ensure its long term attractiveness and sustainability? (1: This aspect has not yet been thought of at all; 2: Some aspects of continuous improvement have been considered, but are not yet implemented; 3: some aspects of continuous improvement are being implemented; 4: some successes of continuous improvements can be demonstrated; 5: continuous improvement is integral to how the facility operates; 6: work on continuous improvement of the facility exceeds expectations)

5) **Strategic Fit:** Can the facility show that it supports work in areas of strategic priority to EPSRC? Does the facility take steps to align itself with EPSRC's balancing strategies? (1: Does not meet the criteria in any way; 2: partially meets the criteria, but with major weaknesses; 3: partially meets the criteria, but there is room for improvement; 4: meets the criteria; 5: meets the criteria and sometimes exceeds expectations; 6: exceeds expectations)

Overall assessment and score: Does the facility meet the assessment criteria? Which areas could be improved? Are any actions recommended to improve the benefits for users and EPSRC from this facility? (1: Does not meet the criteria in any way; 2: partially meets the criteria, but with major weaknesses; 3: partially meets the criteria, but with minor weaknesses; 4: meets the criteria; 5: meets the criteria and sometimes exceeds expectations; 6: exceeds expectations)

Annual Report for EPSRC Mid-Range Facilities – Contract Manager's Commentary

Facility:____

Address: ____

Director:____

Facility Manager:____

EPSRC Contract Manager responsible: ____

Contract Manager's comment on the performance of the facility, including a longer term view.

Change log

Name	Date	Version	Change
Michele Erat	18/07/2014	1.0	N/A
Michele Erat	06/08/2014	1.1	Version after consultation with portfolio managers
Michele Erat	21/01/2015	2.0	Adapted with recommendations from the Capital Equipment SAT
Michele Erat	11/05/2015	2.1	New timelines
Louise Tillman	01/07/2016	3.0	Updated for 2016 with amendments based on feedback from Mid-range Facilities Statements of Need Panel
Simon Crook	17/7/2017	4.0	Updated for 2017
Simon Crook	16/7/2018	5.0	Updated for 2018 to include Sustainability and some amendments to other questions.
Simon Crook	28/08/2019	6.0	Updated for 2019, some minor changes to user questions to give better comparable data on PI and studentships. Increased page limit to 2.