National Research Facility Annual Reporting Template

This Annual Report will be reviewed by the EPSRC National Research Facility High Level Group with any feedback provided by EPSRC. The report and any feedback should be made available to your advisory committee and will also be used within EPSRC by your individual EPSRC contact and the EPSRC NRF lead for information and discussion.

Timeline 2020:

- Reporting Period for this Annual Report: **1st September 2019 – 31st August 2020**
- Deadline for Annual Reports**: 12th January 2021**
- Assessment by Panel**: February/March 2021**
- Feedback to Facilities: **March/April 2021**

Facility Name: XMaS

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

Start/End Dates Start Date: 15th November 2018 through 14th November 2023

Total Funded Award: £6,909,720: split between Liverpool (£3,427,966) and Warwick (£3,481,754)

1) Value Proposition (max ½ page): What is your facility uniquely placed to provide for UK research?

The XMaS beamline has been supporting the UK material sciences community since 1997 and is embedded at the European Synchrotron Radiation Facility (ESRF) in Grenoble, France. Over the period 2015-2021 the ESRF has implemented a €150M facility upgrade, the Extremely Bright Source (EBS) upgrade, bringing its scientific users a first-of-a-kind, low-emittance, high-energy synchrotron light source. To take advantage of this unique source, XMaS has undergone a major upgrade through 2019 and 2020 to deliver a new state-of-the-art facility that fully exploits the capabilities of the new ESRF machine. The rebuilt XMaS beamline will provide a bright X-ray beam over a wide energy range to investigate the structure and properties of the latest generation of functional materials. The XMaS science portfolio continues to evolve and embraces a broad spectrum of scientific disciplines under the generic theme of materials science; these cut across research themes in physics, chemistry, biosciences, healthcare, engineering and energy. XMaS continues to form an integral part of the UK's synchrotron radiation (SR) infrastructure which includes the ESRF and the Diamond Light Source (DLS).

The main objectives of the facility are to provide the UK materials science community with access to a state-of-the-art X-ray facility (source and experimental equipment) and to provide training for early career scientists including postgraduate and undergraduate students in advanced scientific methodologies. XMaS facilitates X-ray characterisation across a range of temporal and spatial length scales and, by the development and use of novel sample environments, enables *operando* studies allowing the correlation between structure and functional properties to be elucidated. Synchrotron studies tend to be collaborative and in the post-Brexit landscape it is essential that UK scientists are able to maintain, develop and nurture links with international colleagues to increase the range, quality and impact of their research. XMaS will play a pivotal role in this; allowing engagement with EU collaborators and providing an additional access point to the ESRF to develop collaborations and instigate innovative metrologies. These partnerships ensure the future competitiveness, resilience and creativity of the UK materials sector which relies on the development, characterisation and exploitation of novel functional materials.

2) Scientific Excellence

In the period covered by this annual report the primary activity was the complete rebuild of the XMaS beamline which was necessary both to take advantage of the ESRF-EBS upgrade but also to update many of the components on the beamline that dated from the original construction phase (1994-97). This rebuild was a phased activity, beginning with the extension of the experimental hutch, followed by the installation of new optics and finally the update of the components in the experimental hutch, including installation of the upgraded diffractometer. Despite the significant delays caused by COVID-19 the rebuild has been completed only 3 months behind the original schedule and the XMaS beamline will resume user operations in January 2021. More details of the rebuild are given in section 12. The combination of a world class photon source, continual innovation in photon techniques, high quality technical and scientific support as well as a strong international user community will maintain the XMaS beamline at the frontier of advanced materials characterisation. The continuing development of novel sample environments ensures a diverse and evolving scientific programme cutting across the challenge themes and complements and strengthens UK-based facilities. The extension of the X-ray energy range to over 40 keV opens up new opportunities for *operando* studies, in which material structure can be directly linked to functionality in realistic working environments.

Although the XMaS beamline was not operational in this reporting period, the off-line facilities continued to operate and scientific output from previous experiments continued apace. A sub-set of the research activity outcomes from the 2019-20 period are presented below, grouped around challenge themes:

Energy research: Research covered photovoltaics, battery materials and catalysis but also issues associated with storage of radioactive materials from the nuclear industry. Performing *operando* experiments enabled by the development of bespoke sample environments allowed new insights into the relationship between structure and functional properties.

A broad range of catalysis research was reported in 2020. *In-situ* extended X-ray absorption fine structure (XAFS) spectroscopy was used in continuous flow reactor cells to explore the reactive behaviour of three catalysts based on Pd-loaded $LaFeO_3$ (ACS Catal. 10, 3933-3944 (2020)) and to study, temporally and spatially, the leaching of Pd from a heterogeneous catalyst caused by K_2CO_3 (Catal. Sci. Technol. 10, 466 (2020)). The chemical and structural nature of potassium compounds involved in catalytic soot oxidation were studied under *operando* conditions at the potassium K-edge (Phys. Chem. Chem. Phys. 22, 18976 (2020)). XANES was used to understand the effectiveness of gallium and tin doped zeolites as catalysts for the conversion of glucose into fructose, mannose and 5-Hydroxymethyl furfural (Appl. Catalysis A, General 605,117798 (2020)). In addition, both *in-situ* and *ex-situ* XANES have explored the ligand environment of single-site Au/C catalysts during acetylene hydrochlorination as a pathway to reduce the environmental impact of this industrial process (Chem. Sci. 11, 7040 (2020)) along with the synthesis of atomically dispersed $PdCl_x/C$ as a model catalyst for H_2O_2 production (ACS Catal. 10, 5928-5938 (2020)).

In functional energy devices, the local structure of the electrodes is critical for device performance. The structure of two naphthylene-capped oligothiophene thin-film field-effect transistor assemblies was studied using grazing incidence X-ray diffraction (Cryst. Growth Des. 20, 6, 3968–3978 (2020)) and new understanding of the effects of surface stress and surface relaxation on the electrochemical reactivity was reported (Current Opinion in Electrochemistry 19,168–174 (2020)).

The solubility, speciation and the local environment of chlorine in zirconolite glass–ceramics, which have been suggested as a potential immobilisation route for chloride contaminated plutonium oxide residues, was studied using XAS (RSC Adv. 10, 32497 (2020)) and the associated press release was picked up in the wider online literature.

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 *operando* conditions.

In soft matter systems, studies have involved nanoconfined ionic liquids (ILs) (Adv. Funct. Mater. 29, 1905054 (2019)) and the structure of the twist-bend nematic phases which exhibit chirality and circular dichroism (Mater. Chem. C 8, 1041-1047 (2020)). High resolution SAXS allowed a robust general design concept for creating bicontinuous cubic phases to be reported (Adv. Funct. Mater. 2004353, 14 p (2020)). XRD studies of bent-core liquid crystals showing a nematic phase based on a five-ring bent-core mesogen were performed to provide complementary information to ^{2}H NMR to unlock the structural properties of the nematic phase and the average mesogen conformation (Crystals 10, 284, 15p (2020)). A new liquid crystalline honeycomb phase was reported, containing highly stretched giant hexagonal cells in a self-assembled liquid crystal *(*Chem. Commun. 56, 62-65 (2020)) as well as novel bolapolyphiles soft-matter cage-like grids with $Pm\overline{3}m$ symmetry (Am. Chem. Soc. 142, 7, 3296-3300 (2020)). X-ray reflectivity has been used to monitor structural changes over a period of 24 hours of supported lipid bilayers embedded with hydrophobic quantum dots (J. Colloid Interface Sci. 562, 409-417 (2020)) and to explore the structure of hybrid nanoparticle-lipid multilayers (Biochimica et Biophysica Acta (BBA) - General Subjects).

Fundamental studies of ferroelectric materials using both scattering and spectroscopic tools to understand the complex interrelationship between their structure and functional properties were reported, much of this work having been undertaken as part of the EMPIR JRP program *ADVENT: Metrology for advanced energy-saving technology in next-generation electronics applications* (2017-2020). XAS studies of dilute Fe-doped $BaTiO_3$ crystals highlight possible new routes for novel ferroelectrics (APL Materials 8, 031109 (2020)). Grazing incidence X-ray diffraction studies of the near surface of a (PIN-PMN-PT) [011] poled rhombohedral single crystals allowed crystallographic strains to be compared with the macroscopic strain as a function of depth under applied electric fields (Journal cover, Crystals 10, 728, 13 p (2020)). The complex relationship between stress/strain and electric field was untangled with our new stress cell that revealed domain-engineered relaxor single crystals that can be dynamically and reversibly driven through a ferroelectric–ferroelectric phase transition across a wide range of frequencies (Appl. Phys. Lett. 116, 222903 (2020)). High resolution diffraction and polarisation analysis elucidated the spin, lattice, charge, and orbital coupling mechanism underlying the multiferroic character in tensile-strained $EuTiO_3$ films. (Phys. Rev. B 101, 180409(R), 6 p (2020))

Proximity-induced magnetization (PIM) has broad implications across interface-driven spintronics applications that employ spin currents with the scaling between the magnitude of PIM in Pt and the temperature-dependent interface magnetization in an adjacent ferromagnet (FM) reported (Phys. Rev. B 100, 174418 (2019)), The PIM in rare earth/transition metal alloy films determined using X-ray magnetic reflectivity *(*Sci. Rep. 10, 9767, 8 p (2020)) highlighted the importance of direct *d-d* coupling in such heterostructured spintronic systems (Phys. Rev. Research 2, 033280, 6 p (2020)). Data was also used in a programme to explore and compare current fitting strategies for magnetic reflectivity at hard energies (Phys. D: Appl. Phys 53, 375004, 17 p (2020)).

Synchrotron X-ray diffraction has also been applied to study the effect of slip additives on the fibre crystal structure of artificial polypropylene fibres (J. Colloid Interface Sci. 571, 398-411 (2020)) and to explore the effects of strain and film thickness on the structural and magnetic properties of Sr_2IrO_4 thin films. (Phys. Rev. B 102, 214402 (2020)). Diffraction was also used to characterize the alloy composition and crystalline surface corrosion of copper alloy Tudor artefacts recovered from the undersea wreck of King Henry VIII's warship the *Mary Rose* giving insight into the conservation treatment applied (J. Synchrotron Rad. 27, 11 p (2020)). This produced considerable interest in the wider online literature with over 20 web-based reports including an article in the telegraph. This research forms one of the case studies that are attached to this report.

In the area of **healthcare**, the mineral–water interface was explored in hexagonal fluoroapatite (0001) using our bespoke sample environment at ambient temperatures (Acta Cryst. B 75, 830–838 (2019)) giving insights into the growth of bone apatite fibres.

It is clear from the above examples that the output from XMaS impacts a wide range of scientific disciplines. The research activities are frequently singled out for inclusion in the ESRF's publications which in the current issue (2019 ESRF Highlights) highlights the work of Dann EK *et al. Structure Selectivity of Supported Pd nanoparticles for Catalytic NH_3 Oxidation resolved using combined Operando Spectroscopy (*Nat. Catal. 2, 157-163 (2019)) and Lilliu S *et al. High-efficiency perovskite–polymer bulk heterostructure light-emitting diodes* (Nat. Photon. 12, 783-789 (2018)).

XMaS activities are widely disseminated in an annual newsletter. The 2019 version was available from Feb. 2020 and circulated widely (>600 copies). A summary of the content of the 2019 newsletter is given below:

- **Empowering young women to pursue careers in STEMM**: *A reflection of 5 years of the XMaS scientist experience competition and its impact on the winners.*
- **Perovskite-polymer bulk heterostructures for efficient LEDs** (Nat. Photon. 12, 783 (2018)): *Demonstration of a high quantum efficient perovskite-polymer blends for the next generation of LEDs with structural information derived from GI-WAXS.*
- **Assessing the solubility of spent nuclear fuel**: *X-ray reflectivity using the offline facility to probe the effect of corrosion of mixed oxide films under H_2O_2 as model systems to understand how spent nuclear fuels can be stored safely.*
- **Unravelling the magnetic proximity effect at interfaces** (Phys. Rev. B 100, 174418 (2019)): *Resonant magnetic scattering to explore the temperature dependent proximity induced magnetism in Pt from a ferromagnetic amorphous CoFeTaB alloy.*
- **Polymerisation rate dictates order and intrinsic strain generation in photo-cured dimethacrylate dental polymers** (Macromolecules 52, no.14, 5377-5388 (2019)): *In-situ study of the structural changes within the polymer matrix induced during curing of dental composites.*
- **Soft rectangular sub-3 nm tiling patterns by liquid crystalline self-assembly** (Adv. Funct. Mater. 28, 1804162 (2018))**:** *Distinct tiling patterns are derived from GI-SAXS experiments from liquid crystals which show a new type of honeycomb structure.*
- **Self-assembly of fluoride encapsulated POSS nanocrystals** (Cryst. Eng. Comm. 21, 710-723 (2019)https://www.nature.com/articles/s41598-018-32425-y): *A temperature dependent study of the ordering of polyhedral oligomeric silsesquioxanes which are cage like nanosized building blocks in which a fluoride ion is encapsulated.*
- **Identification and role of interstitial PdNx structures in the selective oxidation of ammonia** (Nat. Catal. 2, 157–163 (2019))**:** *Operando spectroscopic studies correlating the observed structure of NH_3 during oxidation in the presence of Pd nanoparticles using XANES*
- **Electronic changes in $LiFeSO_4F$-PEDOT during Li-ion battery cycling** (J. Power Sources 418, 84–89 (2019)https://www.sciencedirect.com/science/article/pii/S0008622318310121?dgcid=rss_sd_all): *Fe and S K edge studies of the structure of lithium ion batteries during delithiation as a result of in-situ charging.*

Even though the focus of the beamline staff has been on delivering the XMaS upgrade/rebuild programme, a number of new methodologies have also been developed in this reporting period.

i) Photon-in/Photon-out spectroscopies

Metrology developments in spectroscopy have followed on from proof of principal studies that were carried out on XMaS in 2018. By using a Rowland circle geometry and an analyser crystal, these experiments explored the possibility of studying the electronic structure of matter by methods such as High Energy Resolution Fluorescence Detection (HERFD), X-ray Absorption Spectroscopy (XAS), X-ray Emission Spectroscopy (XES) and Resonant Inelastic X-ray Scattering (RIXS). The preliminary measurements are shown in Figure 1.

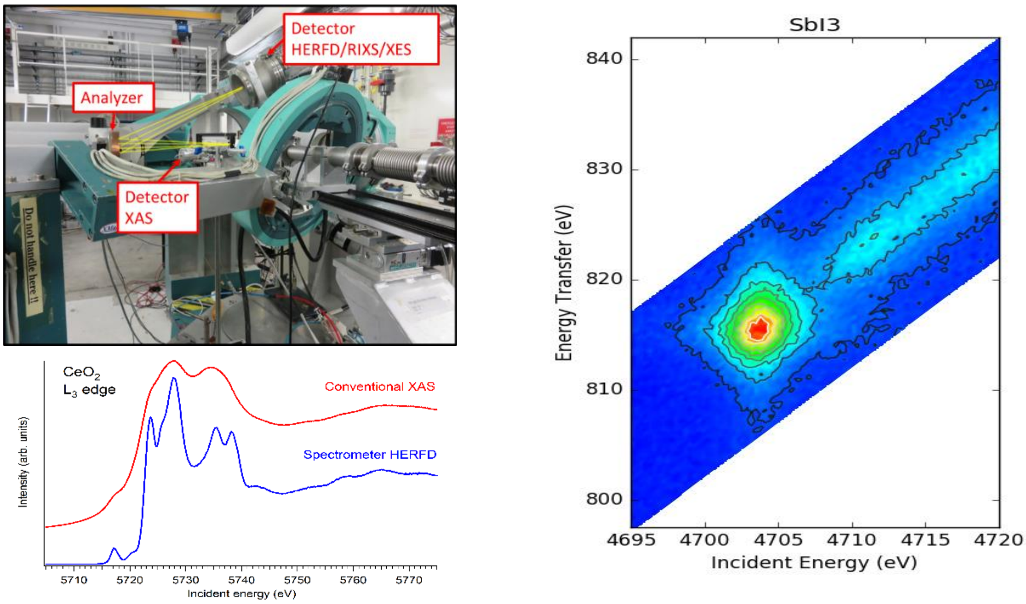

Figure 1: The development of photon in-photon out spectroscopies on XMaS in collaboration with Kristina Kvashnina's Group on BM20 at the ESRF. Exploiting a Rowland circle geometry (left top) for focusing allows both high resolution spectorscopic data (left bottom) and resonant inelastic scattering (right) to be measured.

With the smaller, brighter X-ray beam on the upgraded XMaS beamline such experiments can become more routine. Commissioning of these techniques has continued with the development of data collection strategies and data reduction pathways. Such studies, exploiting the extended X-ray energy range and suite of sample environments at XMaS, provide unique resources to the UK community and complement parallel developments at DLS.

ii) 3D HKL data reduction and visualisation

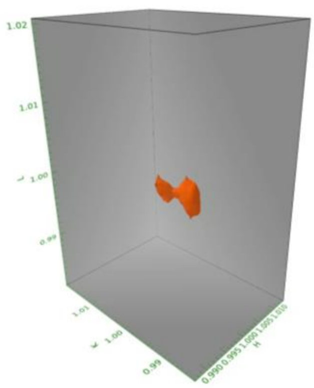

Figure 2: The computed 3D volume of a single scan reduced to HKL co-ordinates.

2D detectors allow an extended region of reciprocal space to be probed either in a single image or built up from multiple images. In both cases, the result of scanning the sample yields data from a defined volume of reciprocal space (figure 2). We have developed a routine to transform the pixel co-ordinates of the detector into the reciprocal space of the sample using the UB matrix, the diffractometer angles, and the detector distance (which is now motorized on the upgraded detector arm). The high number of pixels on the camera can result in large data sets and a simple and fast reduction pathway has been developed to both reduce the data to manageable proportions and to allow rapid visualization of the data. Integrated projections into specific reciprocal space zones are being developed.

iii) Dynamic Studies

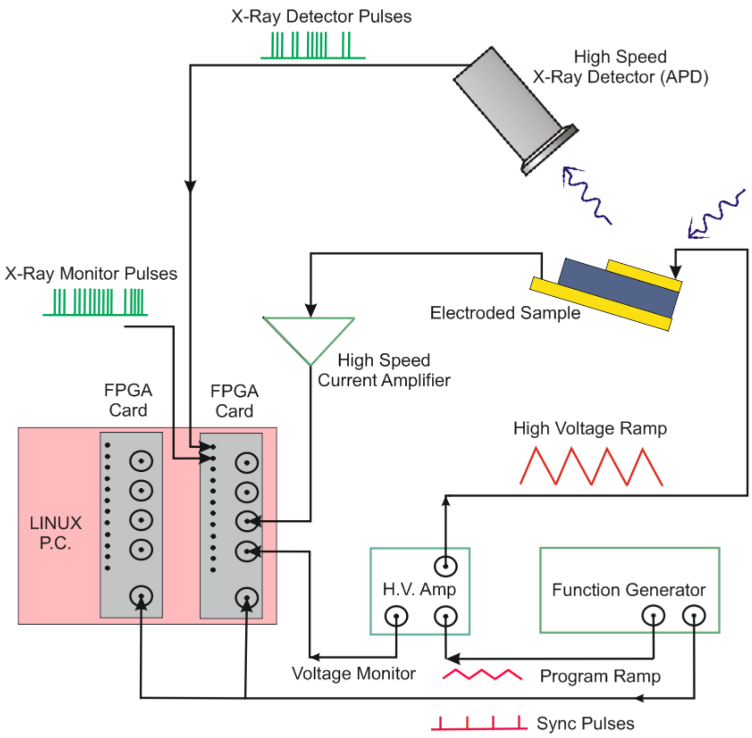

Figure 3: A schematic of the data collection using new FPGA cards which allow X-ray data to be synchronized with functional measurements from the sample.

Much information can be obtained from functional materials if both the structural and functional properties can be measured simultaneously *in-situ* and *operando.* A clear example is in the study of multiferroics where the electrical polarization is intrinsically linked with the structure. As part of the EU-EMPIR funded ADVENT project, new high speed FPGA cards have been incorporated into the data collection protocols (Figure 3) and allow both X-ray data (scattering or fluorescence) to be recorded simultaneously and synchronously with functional data (in this case the electrical polarization, macroscopic strain and/or applied stress). Synchronized data can now be recorded and averaged at frequencies in excess of 100 kHz. The commissioning work to develop these methodologies was done using the offline X-ray source.

iv) New Stress-Strain Sample environment for Ferroelectrics

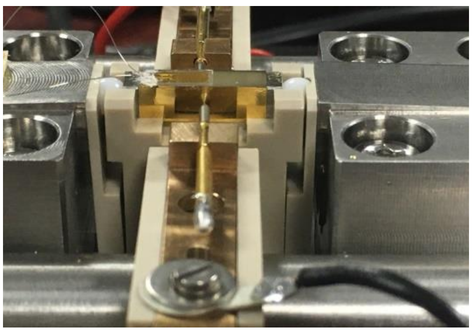

Figure 4: Stress rig with sample mounted. A strain gauge mounted on the upper surface and electrodes contacting platted electrodes on the sides allow functional properties to be measured simultaneously with XRD as stress is applied through the white ceramic balls.

In partnership with Electrosciences Ltd. and also under the umbrella of the ADVENT programme, a new sample environment which can apply uniform stress (200 MPa) to samples whilst simultaneously measuring strain and polarisation has been developed for the studies of ferroelectric samples. Initially this was commissioned and calibrated using a BCR661B Nimonic 75 Alloy Extruded bar before being extended to studies on ferroic materials. X-ray experiments using the offline X-ray source were performed for a PIN-PMN-PT single crystal (Figure 4). Further details are given in the attached *case study.* The sample environment has also been used at the NSLS and Diamond Light Source.

3) Publications

Synchrotron studies are often collaborative in nature with material characterisation being a noted example where many different characterisation tools and approaches are needed to tackle the scientific question. Often, the X-ray data is the "missing link", able to identify or distinguish between competing models. It is difficult to unambiguously state that the results obtained were critical to a particular paper or were important to supporting the conclusions reached.

A full list of the publications is maintained on our website. Herewith, a list of the publications associated with the XMaS NRF published in peer reviewed literature over the past 3 years.

2020

Bikondoa O, Carbone, D,
X-ray Photon Correlation Spectroscopy with Coherent Nanobeams: A Numerical Study
Crystals 10, 766, 16 p (2020). DOI:10.3390/cryst10090766

Cain MG, Staruch M, Thompson PBJ, Lucas CA, Wermeille D, Kayser Y, Berckhoff B, Lofland S, Finkel P,
In-situ Electric-Field Study of Surface Effects in Domain Engineered
$Pb(In_{1/2}Nb_{1/2})O_3$-$Pb(Mg_{1/3}Nd_{2/3})O_3$-$PbTiO_3$ Relaxor Crystals by Grazing Incidence Diffraction
Crystals 10, 728, 13 p (2020) and supplem. DOI:10.3390/cryst10090728

Checchia S, Mulligan CJ, Emerich H, Alxneit I, Krumeich F, Di Michiel M, Thompson PBJ, Mimi Hii KK, Ferri D, Newton MA,
Pd-$LaFeO_3$ Catalysts in Aqueous Ethanol: Pd Reduction, Leaching, and Structural Transformations in the Presence of a Base
ACS Catal. 10, 3933-3944 (2020). DOI:10.1021/acscatal.9b04869

Davies CJ, Mayer A, Gabb J, Walls JM, Degirmenci V, Thompson PBJ, Cibin G, Golunski S, Kondrat SA,
Operando potassium K-edge X-ray absorption spectroscopy: investigating potassium catalysts during soot oxidation
Phys. Chem. Chem. Phys. 22, 18976 (2020) and supplem. DOI:10.1039/d0cp01227k

Dowsett MG, Sabbe PJ, Anjos JA, Schofield EJ, Walker D, Thomas P, York S, Brown S, Wermeille D, Adriaens M,
Synchrotron X-ray diffraction investigation of the surface condition of artefacts from King Henry VIII's warship the Mary Rose
J. Synchrotron Rad. 27, 11 p (2020). DOI:10.1107/S1600577520001812

Dressel C, Reppe T, Poppe S, Prehm M, Lu H, Zeng X, Ungar G, Tschierske C,
Helical Networks of π-Conjugated Rods – A Robust Design Concept for Bicontinuous Cubic Liquid Crystalline Phases with Achiral Ia(-3)d and Chiral I23 Lattice
Adv. Funct. Mater. 2004353, 14 p (2020) and supplem. DOI:10.1002/adfm.202004353

Evans PG, Marks MD, Geprägs S, Dietlein M, Joly Y, Dai M, Hu J, Bouchenoire L, Thompson PBJ, Schülli TU, Richard MI, Gross R, Carbone D, Mannix D,
Resonant nanodiffraction X-ray imaging reveals role of magnetic domains in complex oxide spin caloritronics
Sci. Adv. 6, 8 p (2020) and supplem. DOI:10.1126/sciadv.aba9351

Fox LJ, Slastanov A, Taylor N, Wlodek M, Bikondoa O, Richardson OM, Briscoe WH,
Interactions between PAMAM dendrimers and DOPC lipid multilayers: Membrane thinning and structural disorder

Biochimica et Biophysica Acta (BBA) - General Subjects, Available online 24 January 2020.
DOI: 10.1016/j.bbagen.2020.129542

Geprägs S, Skovdal BE, Scheufele M, Opel M, Wermeille D, Thompson P, Bombardi A, Simonet V, Grenier S, Lejay P, Chahine G, Quintero Castro D, Gross R, Mannix D,
Precise control of Jeff = 1/2 magnetic properties in Sr_2IrO_4 epitaxial thin films by variation of strain and thin film thickness
Phys. Rev. B 102 214402 (2020). DOI:10.1103/PhysRevB.102.214402

Ghilardi M, Adamo FC, Vita F, Francescangeli O, Domenici V,
Comparative ^{2}H NMR and X-ray Diffraction Investigation of a Bent-Core Liquid Crystal Showing a Nematic Phase
Crystals 10, 284, 15p (2020). DOI:10.3390/cryst10040284

Gründer Y, Lucas CA,
Potential-induced structural deformation at electrode surfaces
Current Opinion in Electrochemistry 19,168–174 (2020). DOI:10.1016/j.coelec.2019.12.009

Gubała D, Harniman R, Eloi J-C, Wąsika P, Wermeille D, Sun L, Robles E, Chen M, Briscoe WH,
Multiscale characterisation of single synthetic fibres: Surface morphology and nano-mechanical properties
J. Colloid Interface Sci. 571, 398-411 (2020). DOI:10.1016/j.jcis.2020.03.051

Inyang O, Rafiq A, Swindells C, Atkinson D,
The role of low Gd concentrations on magnetisation behaviour in rare earth:transition metal alloy films
Sci. Rep. 10, 9767, 8 p (2020). DOI:10.1038/s41598-020-66595-5

Krieft J, Graulich D, Moskaltsova A, Bouchenoire L, Francoual S, Kuschel T,
Advanced data analysis procedure for hard X-ray resonant magnetic reflectivity discussed for Pt thin film samples of various complexity
Phys. D: Appl. Phys 53, 375004, 17 p (2020). DOI:10.1088/1361-6463/ab8fdc

Ledendecker M, Pizzutilo E, Malta G, Fortunato GV, Mayrhofer KJJ, Hutchings GJ, Freakley SJ
Isolated Pd sites as selective catalysts for electrochemical and direct hydrogen peroxide synthesis
ACS Catal. 10, 5928-5938 (2020). DOI:10.1021/acscatal.0c01305

Malta G, Kondrat SA, Freakley SJ, Morgan DJ, Gibson EK, Wells PP, Gianolio MAD, Thompson PBJ, Johnston P, Hutchings GJ,
In-situ K-edge X-ray absorption spectroscopy of the ligand environment of single-site Au/C catalysts during acetylene hydrochlorination
Chem. Sci. 11, 7040 (2020) and supplem. DOI:10.1039/D0SC02152K

Newton, MA, Ferri D, Mulligan CJ, Alxneit I, Emerich H, Thompson PBJ, Hii KK,
In-situ study of metal leaching from Pd/Al_2O_3 induced by K_2CO_3
Catal. Sci. Technol. 10, 466 (2020). DOI:10.1039/c9cy02121c

Oozeerally R, Pillier J, Kilic E, Thompson PBJ, Walker M, Griffith BE, Hanna JV, Degirmenci V,
Gallium and tin exchanged Y zeolites for glucose isomerisation and 5-hydroxymethyl furfural production
Appl. Catalysis A, General 605,117798 (2020) and supplem. DOI:10.1016/j.apcata.2020.117798

Patterson EA, Staruch M, Matis BR, Young S, Lofland SE, Antonelli L, Blackmon F, Damjanovic D, Cain MG, Thompson PBJ, Lucas CA, Finkel P,
Dynamic piezoelectric response of relaxor single crystal under electrically driven interferroelectric phase transformations
Appl. Phys. Lett. 116, 222903 (2020). DOI:10.1063/5.0007820

Poppe S, Cheng X, Chen C, Zeng X, Zhang R, Liu F, Ungar G, Tschierske C,
Liquid Organic Frameworks: The Single-Network Plumber's Nightmare Bicontinuous Cubic Liquid Crystal
Am. Chem. Soc. 142, 7, 3296-3300 (2020) and supplem. DOI:10.1021/jacs.9b11073

Ryan PJ, Sterbinsky GE, Choi Y, Woicik JC, Zhu L, Jiang JS, Lee J-H, Schlom DG, Birol T, Brown SD, Thompson PBJ, Normile PS, Lang J, Kim J-W,
Multiferroic behavior in $EuTiO_3$ films constrained by symmetry
Phys. Rev. B 101, 180409(R), 6 p (2020). DOI:10.1103/PhysRevB.101.180409

Scholte A, Hauche S, Wagner M, Prehm M, Poppe S, Chen C, Liu F, Zeng X, Ungar G, Tschierske C,
A self-assembled liquid crystal honeycombof highly stretched (3-1-1)-hexagons
Chem. Commun. 56, 62-65 (2020). DOI:10.1039/c9cc08502e

Swindells C, Nicholson B, Inyang O, Choi Y, Hase T, Atkinson D,
Proximity-induced magnetism in Pt layered with rare-earth–transition-metal ferrimagnetic alloys
Phys. Rev. Research 2, 033280, 6 p (2020). DOI:10.1103/PhysRevResearch.2.033280

Staruch M, ElBidweihy H, Cain MG, Thompson P, Lucas CA, Finkel P,
Magnetic and multiferroic properties of dilute Fe-doped $BaTiO_3$ crystals
APL Materials 8, 031109 (2020) and supplem.DOI:10.1063/5.0002863

Stevenson WD, Zeng X, Welch C, Thakur AK, Ungar G, Mehl GH,
Macroscopic chirality of twist-bend nematic phasein bent dimers confirmed by circular dichroism
Mater. Chem. C 8, 1041-1047 (2020). DOI:10.1039/c9tc05061b

Thornber SM, Mottram LM, Mason AR, P. Thompson, Stennett MC, Hyatt NC,
Solubility, speciation and local environment of chlorine in zirconolite glass–ceramics for the immobilisation of plutonium residues
RSC Adv. 10, 32497 (2020) and supplem. DOI:10.1039/d0ra04938g

Winokur MJ, Huss-Hansen MK, Lauritzen AE, Torkkeli M, Kjelstrup-Hansen J, Knaapila M,
Modeling of Grazing-Incidence X-ray Diffraction from Naphthyl End-Capped Oligothiophenes in Organic Field-Effect Transistors
Cryst. Growth Des. 20, 6, 3968–3978 (2020). DOI:10.1021/acs.cgd.0c00281

Wlodek M, Slastanovs A, Fox LJ, Taylor N, Bikondoa O, Szuwarzynski M, Kolasinska-Sojka M, Warszynski P, Briscoe WH,
Structural evolution of supported lipid bilayers intercalated with quantum dots
J. Colloid Interface Sci. 562, 409-417 (2020). DOI: 10.1016/j.jcis.2019.11.102

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Bikondoa O, Bouchenoire L, Brown SD, Thompson PBJ, Wermeille D, Lucas CA, Cooper MJ, Hase TPA,
XMaS @ the ESRF
Phil. Trans. R. Soc. A 377, 20180237, 12 p (2019). DOI:10.1098/rsta.2018.0237

Blidberg A, Valvo M, Alfredsson M, Tengstedt C, Gustafsson T, Björefors F,
Electronic changes in poly(3,4-ethylenedioxythiophene)-coated $LiFeSO_4F$ during electrochemical lithium extraction
J. Power Sources 418, 84–89 (2019). DOI:10.1016/j.jpowsour.2019.02.039

Burrows CW, Hase TPA, Bell GR,
Hybrid heteroepitaxial growth mode
Physica Status Solidi A 216, 1800600-1-1800600-6 (2019). DOI:10.1002/pssa.201800600

Cherian T, Nunes DR, Dane TG, Jacquemin J, Vainio U, Myllymäki TTT, Timonen JVI, Houbenov N, Maréchal M, Rannou P, Ikkala O,
Supramolecular Self-Assembly of Nanoconfined Ionic Liquids for Fast Anisotropic Ion Transport
Adv. Funct. Mater. 29, 1905054 (2019) and supplem. DOI:10.1002/adfm.201905054

Dann EK, Gibson EK, Blackmore RH, Catlow CRA, Collier P, Chutia A,TE Erden, Hardacre C, Kroner A, Nachtegaal M, Raj A, Rogers SM, Taylor SFR, Thompson P, Tierney GF, Zeinalipour-Yazdi CD, Goguet A, Wells PP,
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Users are encouraged to inform us directly, or through a simple DOI repository on the webpage, of new publications. We also regularly interrogate online repositories such as web of knowledge and/or google scholar to catch missing publications.

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

Impact activities have been severely curtailed by the COVID-19 pandemic which has restricted face to face activities and has been further exacerbated by academic colleagues having to concentrate on moving courses online and reacting to a dynamic and evolving teaching space.

Training Courses and Workshops: In November 2019, the XMaS User Meeting was held at the University of Warwick. Over 50 participants attended during which several users presented work undertaken on XMaS:

- *Understanding and Mimicking the Hierarchical Structure of Human Dental Enamel* [Maisoon Al-Jawad (University of Leeds)]
- *Identification and Role of Interstitial PdN_x Structures in the Selective Oxidation of Ammonia* [Peter Wells (University of Southampton)]
- *The formation of a nanohybrid shish-kebab (NHSK) structure in melt-processed composites of poly(ethylene terephthalate) (PET) and multi-walled carbon nanotubes (MWCNTs)* [Ellen Heeley (Open University)]
- *Structural changes in quasi one-dimensional systems using polarised XAS* [Silvia Ramos (University of Kent)]
- *In-situ grazing incidence diffraction studies of organic thin films* [Andreas Lauritzen (University of Oxford)]
- *Industry Use of Synchrotron Beamlines* [Tim Hyde (Johnson Matthey)]
- *Understanding how to engineer interfacial magnetic proximity effects through X-ray resonant magnetic reflectivity* [Richard Rowan Robinson (University of Sheffield)]
- *An update from the EMPIR ADVENT project - high frequency X-ray diffraction and in-situ applied stress for characterisation of multifunctional materials on the XMaS beamline* [Markys Cain (Electrosciences Ltd)]

The meeting was timed to highlight the first application round for users after the beamline upgrade. The new capabilities of XMaS were presented in detail at the meeting which was also a forum for the discussion of sample environment developments and wider user needs in the future.

Activities to promote the facility beyond its core user base: The 2019 XMaS Newsletter was published in February 2020 and was disseminated widely both online and through hardcopy. With the introduction of GDPR legislation, our contact list is smaller than in the past. Following release, however, many more people registered to receive further updates using the online portal. 600 copies were printed and sent to UK physics, chemistry, and material science departments as well as international partners and researchers. Updates about the XMaS upgrade have been regularly posted on twitter through @XMaSBeam.

Facility staff training and career development: Staff training has been through online courses at the ESRF and the universities which included training in EDI and GDPR. All staff had their annual Personal Development Review which covers all aspects of training and career development.

Public engagement: The XMaS science Gala at the University of Warwick in January 2020 attracted over 600 people to an outreach event targeted at the whole family. Participants could 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, 29/01/20)). Feedback from both the public and exhibitors was extremely positive. It also launched the 2019 edition of the *XMaS Scientist Experience* which attracted over 40 applications from all over the country. The winners have been identified, but the COVID pandemic has meant we have been unable to complete the trip. We are hoping to run the 2020 trip in June 2021 and have been forced to cancel the 2021 Gala and Scientist Experience competition.

Examples of societal & economic impacts: In terms of societal impact, the work on the conservation of the Mary Rose artefacts generated interest in many web-based resources. Following on from previous licensing activities, development work is in progress with Huber Diffraktionstechnik GmbH in the development of monochromator technologies.

5) Cost Recovery

The current award coincided with the beginning of commissioning and upgrade of the facility. For the entire of the reporting period, no user operations have been possible on the synchrotron beamline. No cost recovery has, therefore, been possible.

Future plans and issues

UK access to synchrotron radiation through the ESRF and DLS follows the "free at point of use to the best science" model. The Elsy report, submitted to BEIS in 2017, stress-tested this access mechanism in an independent review of National Large Facilities at Harwell and supported it. XMaS is funded with the same user access model and there is, therefore, limited scope to generate direct cost recovery. The upgraded facility has been designed with increasing efficiency as a key driver, ensuring that we can support more users and therefore be more sustainable by reducing the cost per user/experiment.

Some cost recovery is achieved from commercial licences which generate several k£ per annum which is reinvested into the NRF. Further cost recovery can only feasibly be achieved by direct investment from industrial users. This access mechanism is handled through the ESRF. To date, we have had some limited proprietary access through companies such as Johnson Matthey. We are also working closely with Finden Ltd which is a UK SME specialising in advanced characterisation and expert analysis, deriving structure-activity relationships in functional materials using methods such as X-ray absorption spectroscopy with a focus on the study of catalytic materials and electrodes under operating conditions. Finden is exploring the possibility of purchasing up to ~2 days of facility access time per year.

To facilitate industry access, we will be working with selected industrial partners to create industrial case studies and will produce a targeted newsletter to highlight the new capabilities and opportunities of XMaS in terms of provision for industrial users. This will take some time to develop into an industrial portfolio and we aim to finalise it before any upgrade to DLS to ensure that critical business continuity for UK industries that require access to synchrotron studies can be maintained. We have reserved 2 days per 6 month operational cycle to develop these case studies.

6) Users

Due to the upgrade programme, access to the beamline has not been possible in this reporting period, or since the grant was awarded in November 2018. Some limited access to the offline facilities has been possible but this has been significantly impacted by a combination of safety restrictions, in place during the ESRF EBS construction and commissioning phases, and then COVID-19 protocols. When access was possible, we were able to run remote access experiments at 100% capacity on the offline X-ray facility (figure 5). Several user groups exploited the unique sample environments that have been developed for the synchrotron beamline, but are also compatible with the offline instrument. The limited X-ray flux available on the offline facility means that these experiments typically have long run times (several weeks) which limits the number of experiments and users that can be accommodated but provides an additional resource that is open to the UK materials community. We report below the somewhat sparse user statistics that are derived from the offline facility:

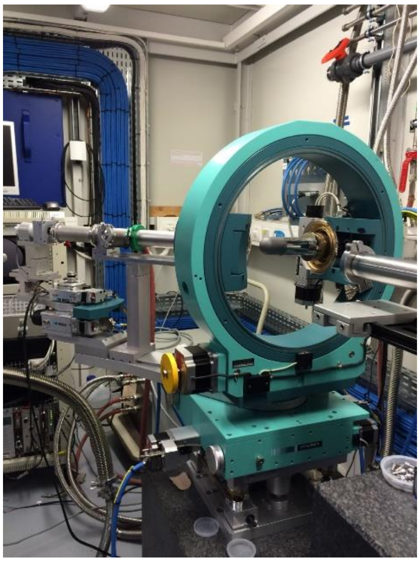

Figure 5: The XMaS offline x-ray source showing a cryostat mounted on the theta axis of the mini Huber diffractometer.

Year	Student	RA	Academic	Industry	Other	No. Repeat	No. unique
2018			1	2	ADVENT	2	3
2019	1		3	1	ADVENT	3	5
2020							

Table 1: User statistics for the offline X-ray Laboratory. Access has included academic user groups, industrial partners, overseas research institutions and the EU-EMPIR funded ADVENT collaboration.

Of these users, 40% were first time users of the offline facility and the student in 2019 was a completely new user of XMaS.

7) User Surveys/Satisfaction

As noted above, user access to XMaS has not been possible over this reporting period. Here we summarise in figure 6 the user feedback from the whole duration of the previous 6-year operational cycle (2012-2018).

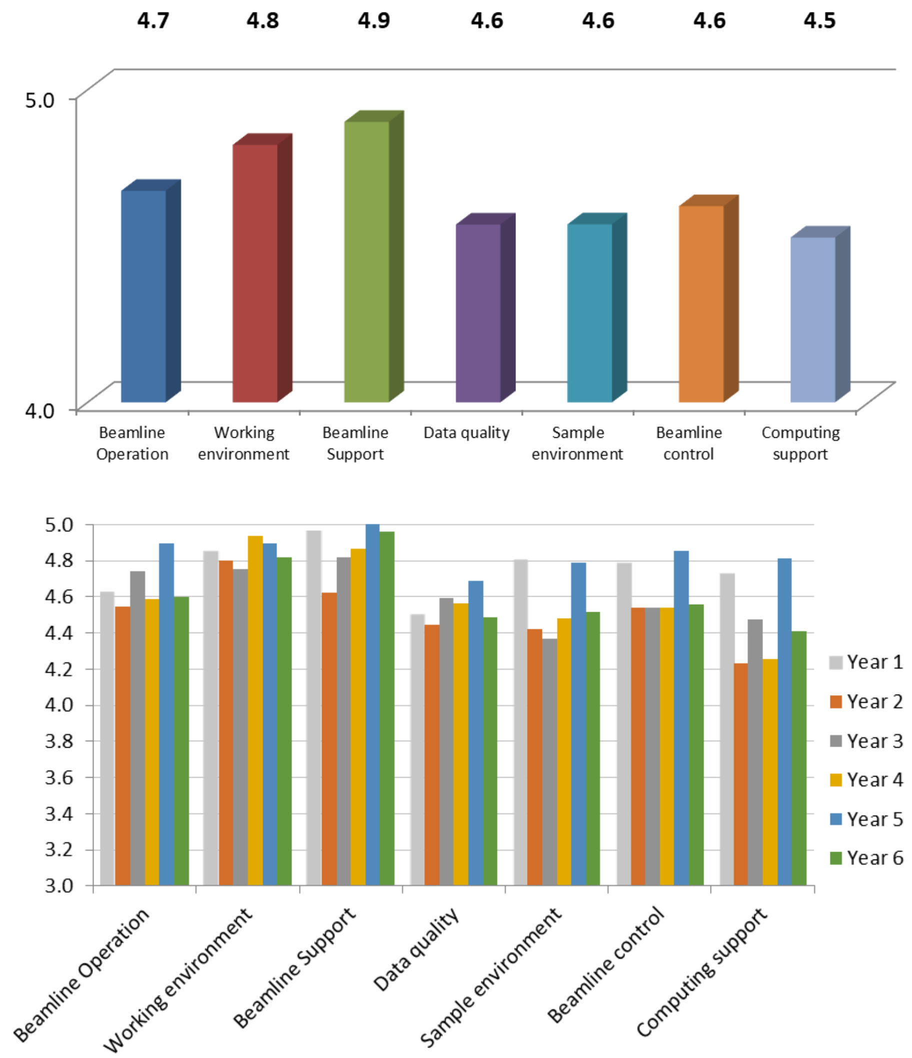

Figure 6: User feedback on facility operations covering the period 2012-2019.

Identified areas for improvement include beamline controls and operations, data quality and computing support. As a result of the upgrade, most experiments will experience an order of magnitude increase in useable flux at the sample, significantly improving data quality for flux limited experiments. The rebuilt stiffer diffractometer ensures higher resolution and stability. These improvements, together with newly acquired detectors and enhancements across the delivery system, coupled with a move to the *psic* control system will significantly enhance the overall beamline operations and support for users. We are currently switching all our motor controllers to the latest ESRF IcePAP standard and improving the computing hardware by installing three new high-performance workstations. The combined effect will be more reliable and smoother controls as well as faster data reduction/visualisation pathways and enhance the user experience. The appointment of two PDRAs, to provide closer and more tailored user support in data management, reduction and processing, has been delayed due to COVID-19 but it is planned to make these appointments as soon as access to the ESRF becomes possible.

8) Service Demand

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. The remaining beamtime, which is available only to the UK community, is allocated through our own review panel process (an independent Peer Review Panel assesses the proposals on scientific merit).

Figure 7 shows the number of proposals received through the two separate access routes as a function of time. We note that despite being offline for over a year and with the added uncertainty on how the ESRF would be operating, a similar number of proposals were submitted compared to previous operational periods. The access calls were unfortunately timed when university research activities and plans had been significantly impacted due to COVID-19 restrictions, a fact reflected by the overall decrease in proposals submitted to central facilities worldwide. Despite the COVID-19 situation there has been a significant number of proposals received through the ESRF route indicating a strong international demand for the use of XMaS.

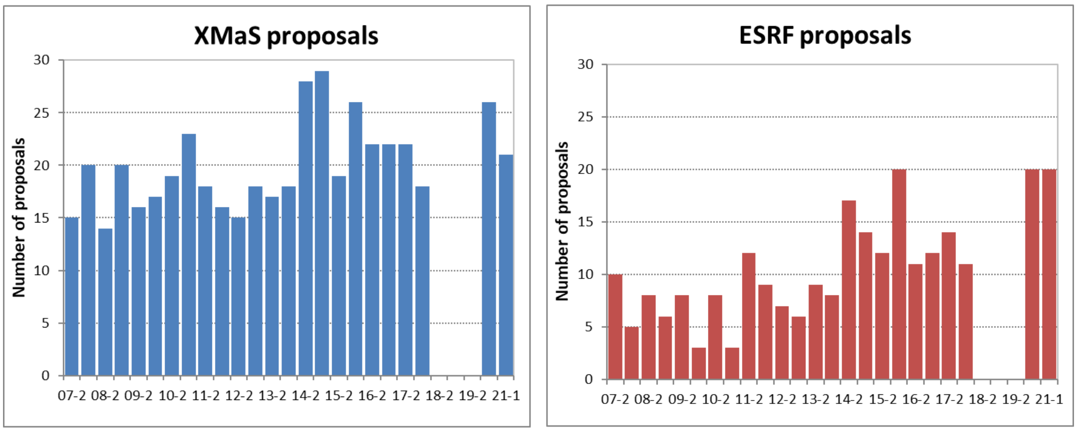

Figure 7: The number of proposals submitted to the XMaS NRF and reviewed by the independent review committees as a function of time. UK based proposals (left) and international applications through the ESRF (right).

Oversubscription rates can be calculated from the ratio of the total number of requested shifts to the total number of shifts delivered for user operations. Historically, using the multi-bunch beam slots in the calculation of oversubscription rates (as is done for the ESRF statistics) the rate is ~2.4 for applications through the XMaS route, although this number drops to ~1.7 if all available operational modes are included (at XMaS we try to schedule all operational modes of the ESRF to give the maximum possible time to users). The international oversubscription rate is steadily increasing and now approaches 4. In these figure 7 we have intentionally not included oversubscription rates for the two latest calls. As experiments have been forced to move to remote access due to the COVID-19 restrictions, only a subset of proposed experiments can be accommodated (those with complex sample environments or sample preparation cannot be done). With operational conditions hugely different and subject to change at short notice it is not a simple calculation to perform and the statistics would be unreliable for these allocation periods.

In addition to the synchrotron beamline, we have developed a range of "offline" facilities including a magneto-transport laboratory for material characterisation under electric and magnetic fields, as well as a micro-focus X-ray source for diffraction, reflectometry and simple SAXS experiments. Over the reporting period, 3 proposals were received for access to the X-ray lab source which utilised all the available 73 days. No further time could be scheduled due to access restrictions to the experimental area during ESRF machine commissioning and then COVID-19 protocols through 2020. We were unable to offer access to the magneto-transport laboratory as staff resources, which are necessary to run this laboratory, were reprioritised to deliver the beamline upgrade.

9) Risks

There has not been a formal risk register for the project to date; the main risks to the project are discussed openly at the 6-monthly Project Management Committee (PMC) meetings. However, as a response to this document a new risk register has been produced and will be reviewed as a standing item at future PMC meetings.

The main identified risks over the reporting period have been the possible impact of Brexit of the movement of people, equipment and consumables to France. Financial risk associated with Brexit uncertainties on the exchange rates has been mitigated by top-loading capital expenditure early during 2018-19, but it remains a risk that needs to be managed. Reputational risk arising from delays to the rebuilding programme due to COVID-19 have been managed with updates and revised operational conditions disseminated to the user community and specifically to any impacted users in a timely manner. This is in line with the procedures that have been taken by many central facilities such as ESRF, DLS etc. Risks are continually reviewed in meetings between the management, beamline staff and the PMC members but will be formalised in the document being prepared.

Regular review of the risk register (at a minimum every quarter, but with the expectation that it will be reviewed monthly) will be conducted by the XMaS management team with the beamline responsible staff member in Grenoble. New risks will be added where needed, and risk scores amended. Where appropriate actions will be identified, and mitigation strategies planned and implemented.

10) Key Performance Indicators (KPIs) and Service Level (SLs) (max. 2 pages)

Since December 2018, the ESRF has been upgrading its magnetic lattice (EBS upgrade programme) and has been shut down with no user operations possible. We have exploited this “dark time” to undertake an extensive capital upgrade and rebuild of the XMaS beamline ready for a planned full user operational cycle in 2021. Limited user operations at the ESRF were expected to resume in August 2020, but the impact of COVID-19 has had an impact on both the machine upgrade and beamline rebuilds. Constrained access to the ESRF site for staff and contractors, combined with local lockdowns and travel difficulties has introduced delays since March. Projects have necessarily had to be completed sequentially and with fewer resources deployed at any given time. The ESRF, however, has been offering very limited, remote access to users since September 2020 and XMaS is now on-track to accept remote users from January 2021. As with other central facilities around the world, users cannot currently travel to the ESRF and only experiments that can be done remotely and handled by the beamline team have been scheduled.

XMaS operates under the following **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.

and **Service Level Agreements (SLAs)**

- 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 full historical record of KPI and SLA data is maintained on our webpage (www.xmas.ac.uk) and is updated quarterly in standard operating periods.

Key Performance Indicators

A) The number of Individual Researchers and University Research Groups visits: zero (0)

Although we have not been able to host users over this reporting period, the track record immediately prior to shutdown shows that we have increased operational efficiency over the past few years increasing both the number of user visits and number of experiments performed on the facility (Figure 8).

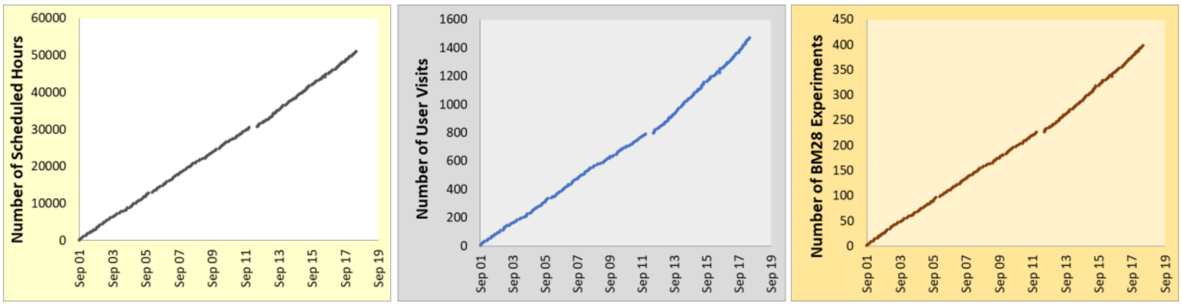

Figure 8: Historical performance metrics for XMaS including the number of scheduled hours (left), the number of user visits to the facility (centre) and the number of individual experiments performed on the beamline (right).

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

C) Uptime of the facility: zero (0)

Historically the uptime of the facility exceeds 95% with downtime primarily associated with storage ring failures. Uptime of the offline facility was 99% over this reporting period.

D) The number of research outputs: Over the reporting period there were a total of 30 published papers. The total number of published XMaS papers has now exceeded 450 (figure 9).

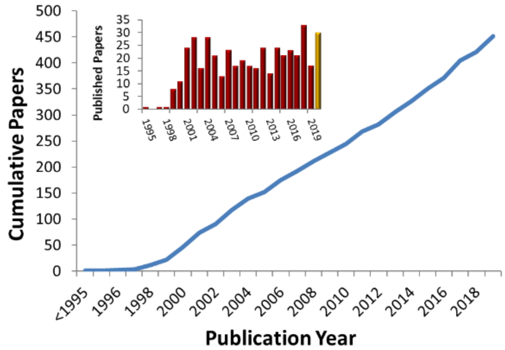

Figure 9: Publications from XMaS in peer reviewed journals. Over the reporting period 30 papers were published with a cumulative total of over 450.

Although publication rates are relatively consistent there has been a steady shift to output in high impact journals with nearly a third of the output 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 members of the XMaS staff. There are also many conference and seminar presentations which are more difficult to capture. Prof. Andy Dent (Deputy Science Director at DLS) noted in the last ESRF quinquennial review that "on similar beamlines at DLS, which usually have weeklong experiments, the typical publication rate is somewhere between 12 and 20 high quality publications per annum."

Service Level Agreements

SLAs have all been met within the uncertainties associated with COVID-19. We are the first beamline at the ESRF to have an equality, diversity and inclusivity statement. Due to COVID-19 there has been no opportunity for workshops and meetings in 2020 but a User Meeting was held in at the University of Warwick in November 2019 and the annual newsletter was disseminated in February 2020. Regular updates on twitter have kept the user community up to date with the changes on the beamline. We have liaised with users about the changing needs and requirements for beamtime and access from January 2021 informing users of any changes as soon as we are aware. The situation remains constantly under review and is subject to any changes in French law or directives from the ESRF safety office.

11) Links

As an integral component of the UK Synchrotron Radiation infrastructure, XMaS naturally has strong links with the Diamond Light Source (DLS) and has been operational throughout the development of the DLS beamline portfolio. DLS executives are fully cognisant of the capabilities of XMaS with Prof. Andy Dent (Deputy Science Director at DLS) reviewing the facility as part of the latest quinquennial review conducted by the ESRF. A DLS science group leader (Prof. Chris Nicklin) sits on our Project Management Committee (PMC), along with Prof. Sean Langridge, the head of the ISIS Diffraction and Materials Division. XMaS directors likewise sit on and have chaired DLS review panels and Prof. Tom Hase currently is chair of the DLS Science Advisory Committee. There is a free flow of information about current capabilities and opportunities for collaboration. Access to XMaS is already part of the DLS mitigation strategy for any future DLS upgrade and its concomitant dark period. Beyond Diamond, XMaS also has developed strong links with sectors 4 and 6 at the Advanced Photon Source (Argonne National Laboratory, US) over many years, developing sample environments and metrologies in magnetic and high-resolution diffraction. XMaS users were granted some access to sector 4 during the recent ESRF shut-down and XMaS looks forward to reciprocating and hosting some APS users over the next 18 months (during the planned APS upgrade). We are also developing close contacts at beamline 4 at the National Synchrotron Light Source-2 (Brookhaven National Laboratory, US). The engagement with other facilities is built on long-standing collaborations and mutual trust and support. We fully expect this to continue and develop further in the years ahead.

XMaS has been working closely with key stakeholders at the UK Catalysis hub (Research Complex @Harwell) and Faraday Institution to provide sample environments that exploit the unique ability to access both tender (2-4 keV) and hard (20-40 keV) X-ray photons interacting with the same sample volume. The focus here is on developing sample environments for *in-situ* and *operando* experiments which are crucial for developing the understanding of material function in the working environment that drives advances in catalysis and battery technologies. Prof. Andy Beale (UK Catalysis Hub) sits on our PMC and is also helping to develop our industrial interactions. New PDRA appointments to XMaS (delayed by COVID-19 but to be appointed soon) will focus on providing new data reduction and analysis pathways that will benefit, in particular, XMaS users who require substantial assistance with data analysis methodologies. By working with multiple groups these PDRAs will enable the sharing of best practice and help to facilitate and grow the non-expert user community.

Of course XMaS benefits tremendously from being part of the ESRF and based at the European Photon and Neutron (EPN) campus in Grenoble, France. The EPN campus is an international science hub hosting the ESRF and the world's most intense neutron source, the ILL, along with joint partnerships. It hosts a staff of 1500 people, including 500 scientists or postgraduate students, and typically welcomes more than 8000 guest researchers every year. There is strong interaction between XMaS and the ESRF. Prof. Chris Lucas has sat on several beamline review panels, ESRF upgrade steering groups and currently chairs one of the proposal review panels. The XMaS beamline scientists engage in day-to-day interaction with the broad EPN international scientific community, for example benefiting from the technical expertise both at the ESRF and the ILL in the development of novel sample environments. Users to XMaS become part of this international community often developing new scientific ideas that lead to collaborative projects involving scientists from across Europe and beyond. These interactions are difficult to quantify but are of crucial importance.

12) Improvements and future plans

In 2019-20 the ESRF completed the final phase of its upgrade programme with the installation of an ultra-low emittance storage ring - the extremely brilliant source (EBS) lattice (www.esrf.eu/about/upgrade). The shutdown of the ESRF enabled us to perform a complete upgrade of the instrumentation on XMaS to the current state-of-the-art whilst matching the beamline to the characteristics of the new ESRF source. Post EBS, the beamline will have more than an order of magnitude gain in usable flux for most experiments with a smaller focal spot and access to higher X-ray energies. Following user consultation, in workshops and meetings prior to the beamline upgrade, it was clear that the increasing demand for *operando* and *in-situ* studies meant it was essential to access the higher energy region of the available X-ray spectrum. Thus, a high field (0.85 T) "short bend" source was selected from the options made available by the ESRF. This new source is positioned in the storage lattice some 2.75 m upstream of the current position necessitating a new toroidal focusing mirror. In fact, two mirrors are needed to cover the X-ray energy range which now runs from 2.1 keV to a cut-off at ~40 keV (a significant increase from the previous cut off energy at ~15 keV). As much as possible of the original beamline infrastructure was retained and, with the addition of some safety essential components and a fully commissioned cryo-cooled monochromator, the optics hutch was completed in July 2020 (Figure 10).

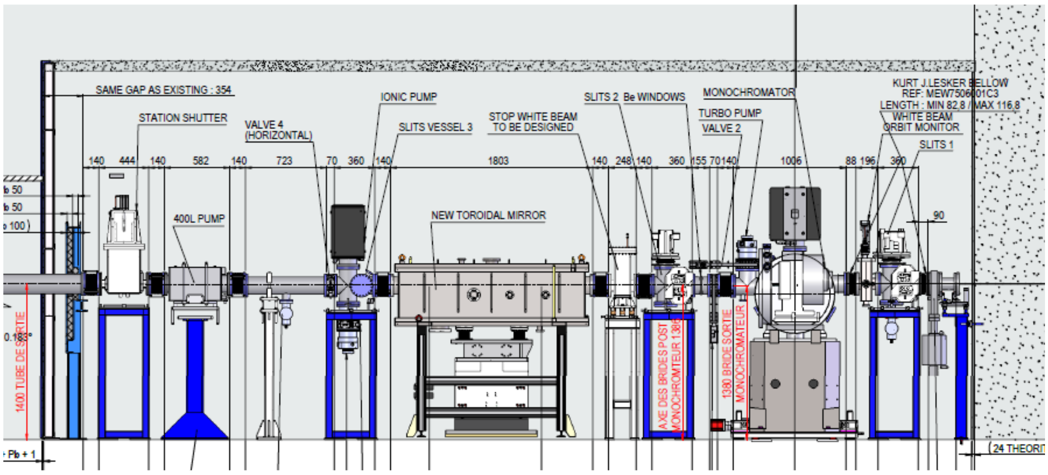

Figure 10: Schematic of the new optics hutch.

The upgraded beamline has retained the three operational modes; focused monochromatic, unfocused monochromatic and white beam (although the latter still requires ESRF safety approval). A change in the mirror angle, and a lateral translation of the beam required a complete redesign of the incident beam path from the optics hutch to the diffractometer. This is shown in Figure 11:

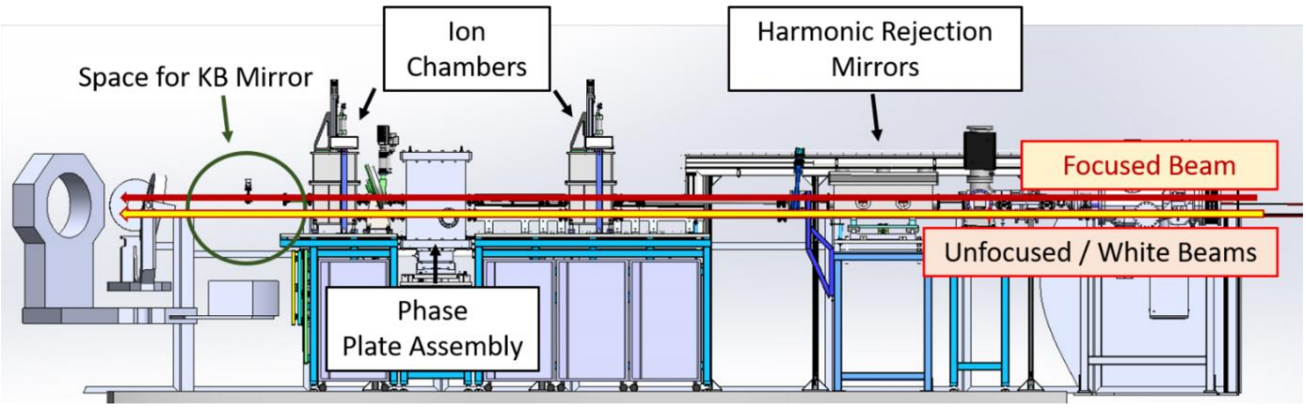

Figure 11: The experimental hutch design.

Extra redundancy, resilience and capabilities have been built into the system including beam position monitors, calibration foils for spectroscopy, ion chambers, attenuation boxes that cover the new energy range, as well as an upgraded phase plate assembly and new harmonic rejection mirrors. The experimental hutch infrastructure was completed at the end of August 2020. Figure 12 shows the “before [November 2019]” and “current [November 2020]” images of the optics and experimental hutches after the extensive rebuild of the beamline that took place in 2020.

Figure 12: Optics hutch (left) and experimental cabin (right) before (top) and after (bottom) the extensive rebuild of the beamline.

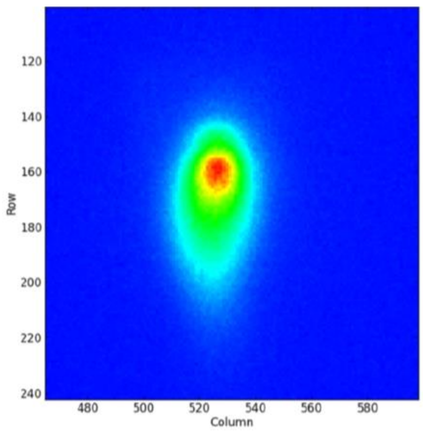

Figure 13: An image of the beam at the sample position at 8 keV. The beamsize is 70 μm in the horizontal direction and slightly larger at 140 μm in the vertical.

The new mirrors and cryo-cooled monochromator now produce a focused monochromatic X-ray beam at the sample position of some 70 x 120 (H x V) μm^2 under normal conditions (figure 13), but which can be further optimised down to 60 x 90 μm^2, or slightly better. This new beam is significantly better focused than the old 300 x 600 μm^2 beam. The new beamsize is independent of energy across the entire 2 to 40 keV range and commissioning is ongoing to ensure that when the energy is changed, the beam position remains unchanged.

Since September 2020, the beamline staff have been commissioning various new capabilities, testing controls and benchmarking routine methodologies including reflectivity, diffraction and spectroscopy at both low (3 keV) and high (40 keV) X-ray energies. This has necessitated commissioning and testing the 2D detectors which include a Lambda CdTe and Pilatus3 1M. The control software and hardware are fully interfaced and operating smoothly which has allowed us to switch the data format to the facility standard hdf5. To ensure findability, accessibility, interoperability, and reusability (FAIR) of all XMaS produced data we will implement the ESRF data policy and ensure that each experimental data set is allocated a DOI reference number to be quoted in publications.

The workhorse of the XMaS beamline is the diffractometer, which is used in nearly all experiments. From the end of 2019 through to the end of the summer of 2020, the old diffractometer was completely renovated, with gears replaces and axes re-aligned. This has returned the sphere of confusion to the original specification of <30 μm. A major upgrade has been the stiffening of the detector arm so that it does not distort when heavy 2D cameras are mounted, ensuring high accuracy and stability are maintained. To further enhance the user experience and operability, the additional motorised slides allow the camera positions to be moved. The upgraded diffractometer with the new double 2θ axis is shown in Figure 14.

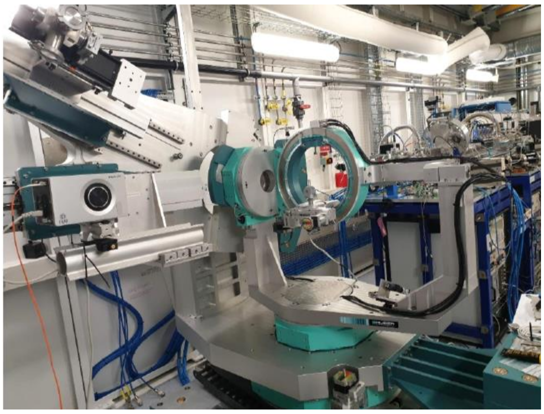

Figure 14: The refurbished Huber diffractometer with new double 2θ axis.

At the time of writing, the commissioning data shows that we can reproducibly change energy and switch between mirrors whilst retaining a small beam size. Further studies are needed to confirm the stability of the beam position, but at the moment its position remains within 50 μm as the energy is changed and this is expected to improve still further over the coming months.

COVID-19 restrictions have, unfortunately, meant that only one staff member can work on the beamline at a given time resulting in commissioning works being slower and projects having to run sequentially, rather than in parallel. These restrictions have required us to prioritise which capabilities can be delivered on day 1 of user operations. Further commissioning is required on the phase plate assembly for user experiments in March 2021 as well as the gas handling and control system which is built and in-place but not yet operational. Likewise, modification to an extension rail that allows for simultaneous SAXS/WAXS studies has been delayed by 3 months. Further optimisation and efficiency gains will continue to be realised through 2021. **Commissioning over the past few months has shown that simple user operations can begin in January 2021**. **This is testament to the outstanding effort put in by the beamline staff under extremely difficult conditions.**

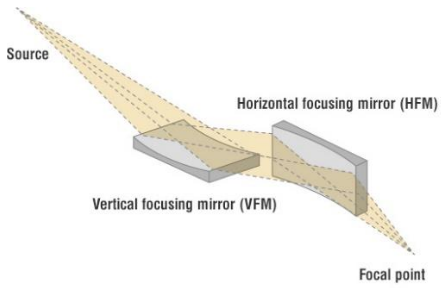

Figure 15: A schematic of a KB mirror which focuses a 2D divergent source using a pair of elliptical mirrors.[i]

One of the key issues to emerge from user consultations was the demand for micron-sized X-ray beams for both *in-situ* and *operando* studies to allow detailed investigations on spatially selected sample volumes. The upgraded beamline can provide small beams using high precision slits, but vibrations result in instabilities in the beam shape and fluctuations in the intensity. We used the recently announced EPSRC capital call to bid for an add-on focusing optic, in addition to the main mirror, to focus a stable beam onto the sample position whilst retaining as much of the flux as possible and ensuring that the beam retains its defined size as the energy is changed. The only choice for such an optic is a Kirkpatrick-Baez, or KB mirror. As shown in Figure 15, this is a 2D focusing mirror system consisting of separate vertical and horizontal mirrors of an elliptical shape. They allow beams to be created down to submicron sizes with an efficiency of 80%. Suppliers will be sought through a tendering exercise in January 2021. Coupled with additional capital items for sample environments and increased resilience to mechanical failures of core equipment, the upgrades to the infrastructure along with the new data visualisation/reduction pathways to come ensures that XMaS is well positioned and sustainable for the long-term. The investments made will ensure internationally competitive science can be delivered (as clearly demonstrated by the high number of international proposals at the last two public calls) and provides enhanced opportunities to the UK materials community for state-of-the-art material characterisation studies.

13) Website

The NRF's website is www.xmas.ac.uk. The webpage will be refreshed and restructured during 2021 to reflect the changes on the beamline and the new capabilities and will include a new section with clearer signposting dedicated to industrial users and access exploiting the industrial case studies.

Web analytics are provided by the host server at the University of Warwick. Excluding search engines, the website receives on average 77 page views per day. The views over the reporting period are presented in Figure 16 below:

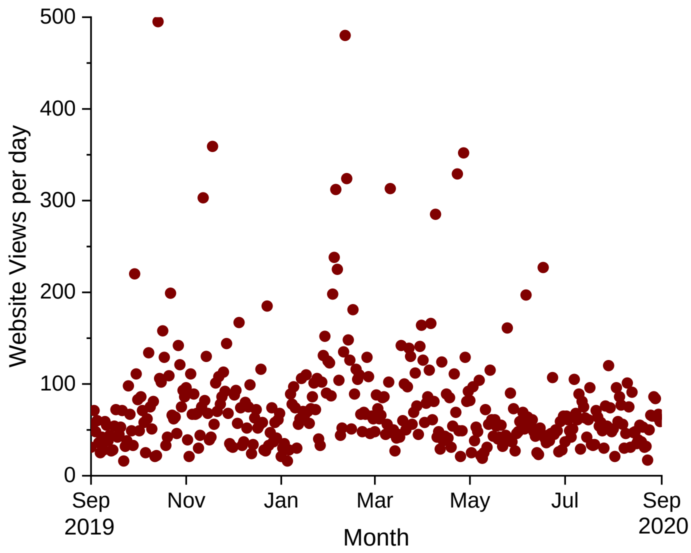

Figure 16: Distinct website views per day over the reporting period.

14) Case Studies

2 Case studies have been prepared and they are:

1. ADVENT: Metrology for advanced energy-saving technology in next-generation electronics applications.
2. Surface condition of artefacts from King Henry VIII's warship the *Mary Rose*.

A further case study is in preparation:

A. Using Zirconolite glass-ceramics for immobilisation of UK nuclear wastes.

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[i] Image taken from https://www.j-tec.co.jp/english/optical/optical-products/kb-mirror/ [July 2020].