XMaS

NEWSLETTER 2021

CONTENTS

On the cover: 4 T superconducting magnet mounted on the refurbished double 2θ arm diffractometer.

The new XMaS beamline!

XMaS has been significantly impacted by the COVID pandemic over the past two years with users and staff having to navigate the constantly changing French and UK rules. However, despite some fluctuations, the situation has steadily improved throughout 2021 and now we can look forward to 2022 and beyond with more confidence.

We were able to begin a restricted user programme on the new, refurbished beamline from January 2021. Operations were limited to remote access with users being able to control and manage their experiments from home. This obviously limited what we could offer, but the beamline team were magnificent in supporting these experiments, mounting samples, setting up measurements, making sure there were no catastrophic hardware collisions, etc. As directors we would like to extend our thanks to the team who ensured that we could support the user community during the various waves of the pandemic. We would also like to thank you, the user community, for your patient understanding and for being flexible with scheduling even if that meant last minute changes and difficulties in securing samples. We had just started to welcome users back to the beamline before Christmas, when the Omicron wave struck and travel to France became impossible again. At the time of writing, we are back to running a full user programme with *in-situ* and *operando* experiments back to the fore. Fingers crossed we are set fair for the future.

The pandemic has not limited our commitment to update and improve the facility. You will see the features of the newly upgraded beamline described on pages 3 and 4 along with a panoramic view of the new delivery systems. We are pleased to announce that we will also be installing a KB system in time for the April 2023 beamtime call. We are in the final design review stage. The system will be mounted on the unfocused/white beam and allow for a tuneable beam size down to a few microns.

In Staff news, Sarah Jarratt has taken over the administrative duties at the University of Warwick from Natacha Borrel who returned to France. We wish her all the best for the future. Sandra Beaufoy also retired in 2021 after supporting the XMaS project for over twenty five years. Thanks Sandra! We are also delighted to welcome Dr Edgar Gutierrez Fernandez to the onsite team as a PDRA with expertise in SAXS. He will be helping users with data collection and reduction, developing new analysis software tools to support user experiments. A second PDRA position is under offer. This PDRA will support users in the area of spectroscopy.

We were honoured to welcome delegates from the British Embassy in France (@UKinFrance @UKSINet) during a tour of the EPN Campus in November 2021. Theo Rycroft, the Minister and Deputy Head of Mission and Sara Gill, the Science & Innovation Policy Officer were given a tour of the beamline (Fig. 1). It was a great opportunity to highlight the importance of UK activities at the ESRF and the importance of maintaining a strong UK presence in European Synchrotron Science.

The Embassy visit also enabled us to highlight our outreach activities and the role that XMaS plays in supporting the UK materials community and the wider public. We have launched the 7th edition of the XMaS Scientist Experience [1] and have had over 50 applications from students across the UK. The quality and enthusiasm of the students is clear to see, and we hope that a trip to XMaS and the ESRF encourages them to pursue careers in STEM. The scientist experience and all our outreach activities are disseminated through our new XMaS twitter account for outreach activities @XMaSScientist.

As we begin to see the end of Covid restrictions, we are also hoping to increase the face-to-face engagement with the user community and find out what you need and want to see developed on the beamline. We are working on setting up a User meeting to be held in the summer of 2022. Further details will be highlighted through the XMaS website and twitter feeds, so please check regularly.

Finally, we are continuing to ensure that user access to the facility accommodates all users. We have funds to develop new sample environments or techniques, so please get in touch if you would like to work with us in developing any new capabilities or software. We are also looking at implementing an access route for Block Allocation Group (BAG) proposals at XMaS similar to those on B18 @DLS. This will be covering the fields of spectroscopy and catalysis. We look forward to seeing you at the beamline in the near future!

Fig. 1: (from left to right) Sara Gill - Science & Innovation Policy Officer, Francesco Sette - DG of the ESRF, Theo Rycroft - Minister & Deputy Head of Mission and Tom Hase - University of Warwick and co-director of the XMaS project. Credit C. Argoud @ESRF

Malcolm Cooper, Yvonne Gründer, Tom Hase and Chris Lucas

[1] www.xmas.ac.uk/impact/outreach/

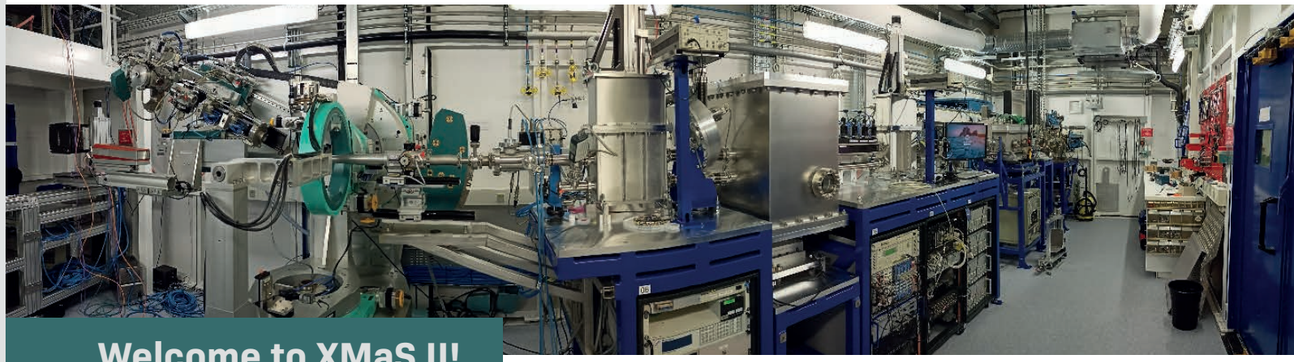

Fig. 2: Panoramic view of the new experimental hutch.

Welcome to XMaS II!

The first X-ray beam from our new source entered the experimental hutch in September 2020. A 3-month commissioning phase to align and calibrate the beamline followed. We used this time to also pin down and fix various issues that arose from dealing with an entirely new beamline and source characteristics; e.g. the higher energy source required the actuator cables inside the monochromator to be replaced with added radiation shielding. The facility resumed user operations in January 2021. However, due to Covid rules this access was limited to remote or mail-in modes for the first half of the year.

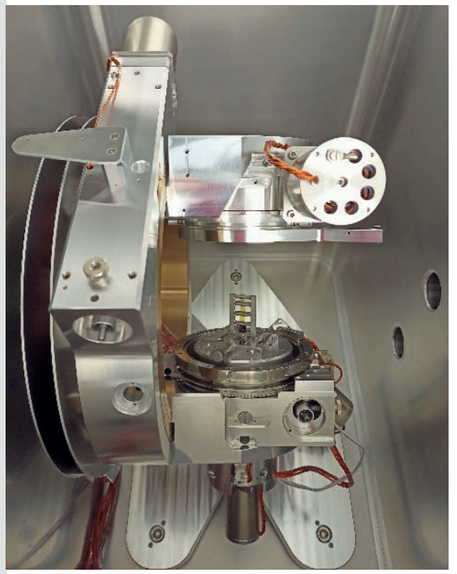

Fig. 3: Inside view of the new in-vacuum phase-plate assembly showing the 4 crystals mounted on top of the flipper.

With the higher magnetic field of the new XMaS source and the extended energy cut-off of the new focusing mirrors, the monochromated beam contains more harmonics than prior to the upgrade. To remove them the original harmonic rejection mirrors were replaced by two silicon mirrors with Pt, bare Si and Cr coatings. The chamber itself was also modified to allow the unfocused beam delivery system to pass underneath.

The phase-plate assembly mounted in a custom-built chamber (Fig. 3) has been operational since mid-2021 in a basic mode. The flipper mechanism for fast helicity reversal is yet to be fully commissioned but will be available to user soon.

An in-vacuum wheel containing up to 30 reference elemental foils (Fig. 4 & 5) is now permanently mounted for energy calibration purposes. Two vacuum vessels are located upstream and downstream of the filter wheel (Fig. 4), with each containing an ion chamber (IC), which can be dosed with different gases. These are now an integral part of the beam delivery. The in-house developed system was designed such that the ICs can be deployed in either the monochromatic focused or unfocused beams and potentially in the white beam when we obtain safety approval for such experiments. The ICs can also be fully retracted for low energy experiments. The automatic gas handling of the ICs and sample environments has necessarily been delayed as the gas delivery system needed to be rehoused due to the new KB mirror project (see article page 5).

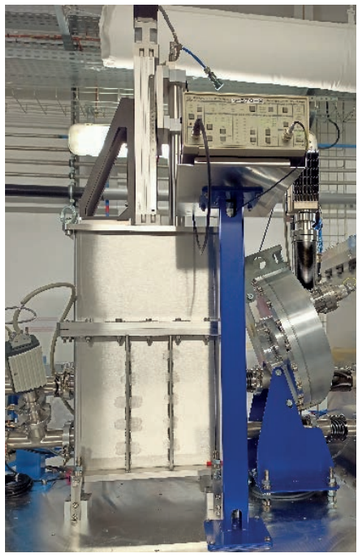

Fig. 4: Filter wheel (right) and retractable in-vacuum ion chamber (left) with current amplifier (middle).

The facility has additional detectors. The new Pilatus3 1M can be mounted on the upgraded SAXS rail (Fig. 6) and positioned over an extended distance ranging from 0.4 up to 2.6 m away from the sample thus facilitating studies down to 0.013 $Å^{-1}$ (~50 nm in real space). In addition to the capability provided by the refurbished double 2θ arm diffractometer, the new configuration offers more opportunities for future studies combining (GI)SAXS/WAXS and XRR.

Finally, a summary of the new specifications and capabilities of the beamline along with the available sample environments are summarised on page 4.

CHANGES TO AND NEW SPECIFICATIONS OF THE BEAMLINE

- Operational energy: 2.035 – 41 keV.
- Double focusing mirror system with Cr (~2 – 21 keV) and Pt (~2 – 41 keV) coatings.
- 50 µm (H) x 80 µm (V) spot, i.e. ~100 times smaller than before.
- Harmonic rejection mirrors with three coatings (Pt, bare Si and Cr stripes).
- Wider suite of detectors (Pilatus3 1M, Lambda 750k CdTe) as well as the previously available APD, Vortex, 150 mm² SDD Ketek, Pilatus3 300K, Maxipix and MAR165 CCD detectors.
- Retractable in-vacuum ion chambers allowing rapid switching between low energy (fluorescence yield) and high energy XAS (transmission) setups.
- In-vacuum filter wheel containing up to 30 reference foils.
- In-vacuum phase-plate chamber with 4 crystals permanently mounted to produce circularly polarised X-rays between 2.4 and 13 keV.
- Refurbished diffractometer with a double 2θ arm allowing rapid switching between measurements with point detectors (Vortex, APD) – e.g. high resolution XRD or HERFD with an analyser crystal, resonant magnetic reflectivity and scattering with polarisation analysis, – and techniques using 2D detectors e.g. (GI)-WAXS, time resolved XRD.
- Upgraded SAXS capabilities with the Pilatus3 1M and extended Q-range down to ~ 0.013 Å^{-1} with a longer SAXS rail of ~ 2.6 m.

AVAILABLE SAMPLE ENVIRONMENTS

- Temperature ranging from 1 to 700 K (cryostats, cryofurnace) combined with magnetic field (between ±0.01 T and ±4 T) and/or electric field (up to ±10 kV). Temperature up to 1000 K on request.
- Ball chamber with vacuum or helium atmosphere, flow cell and sample changer (for powder and ionic liquids) for XAS studies between 2.035 and 41 keV.
- Linkam Single Cell Calorimetry stage (DSC600) and 1.7 mm Ø Capillary system (HFS600-CAP) both with controlled temperature between -195°C and 600°C for SAXS and WAXS.
- Potentiostat for electro-chemical studies.
- MASDAR chamber for GIWAXS with temperature control up to 150°C.

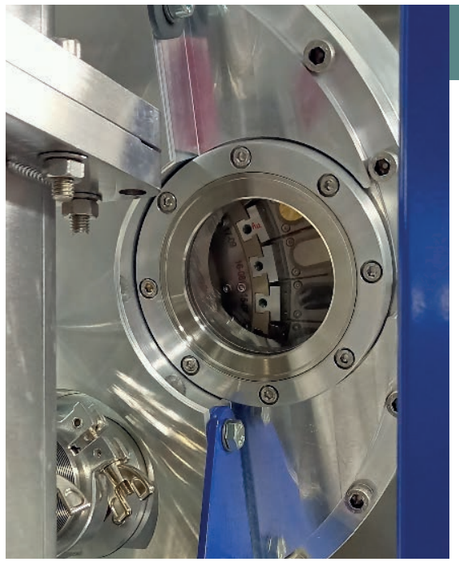

Fig. 5: Filter wheel with up to 30 reference foils for energy calibration purposes.

(GI)-SAXS: (Grazing Incidence) Small Angle X-ray Scattering

(GI)-WAXS: (Grazing Incidence) Wide Angle X-ray Scattering

HERFD: High Energy Resolution Fluorescence Detection

XAS: X-ray Absorption Spectroscopy

XRD: X-ray Diffraction

XRR: X-ray Reflectivity

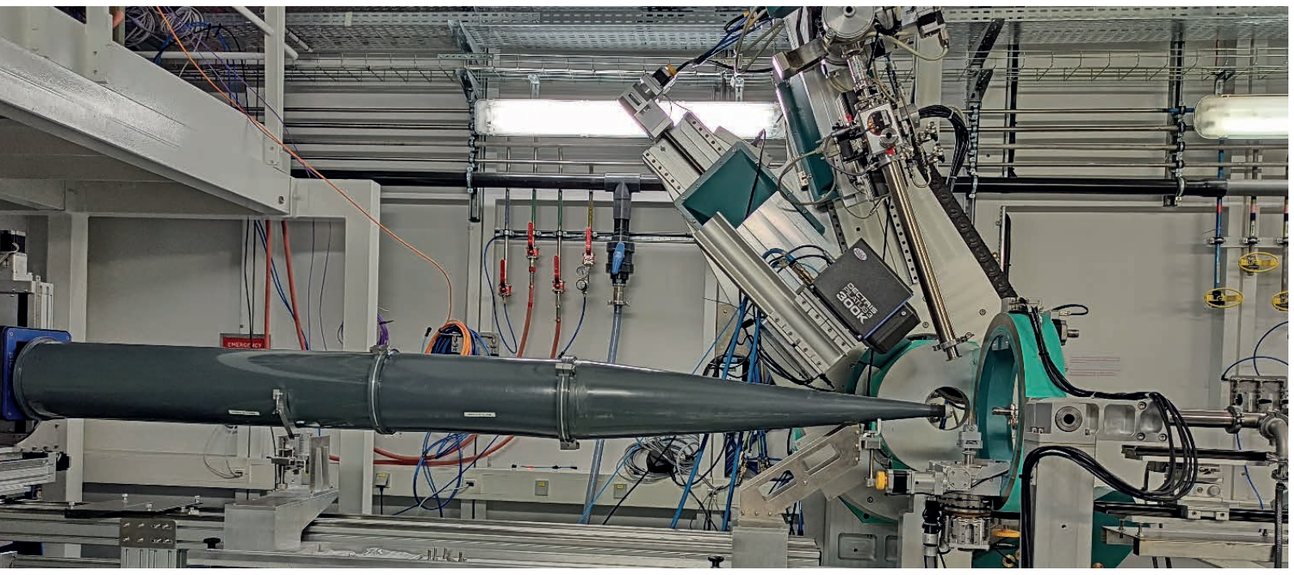

Fig. 6: Extended SAXS setup allowing simultaneous SAXS and WAXS combined with XRR.

KB mirrors

A tender to design and build a KB mirror assembly was awarded to IDT [1] in the spring of 2021. The system is being designed for operations in the monochromatic unfocused beam (MUB) and white beam (WB) paths.

The KB system consists of two silicon mirrors with three stripes (Pt, Rh and bare Si) to cover an energy spectrum between 4 and 25 keV. The whole assembly will be mounted inside an in-vacuum chamber.

The current design is shown in Fig. 7. The in-vacuum chamber can be positioned into the beam and retracted to its park position by means of a circular rail track. This approach minimises constraints on the use of the monochromatic focused beam (MFB). If space is not required for sample environments, the KB mirror can be retracted from the beam path to allow the MFB to pass unimpeded even whilst the KB mirror chamber remains in place. The exact positioning of the system has been designed to ensure that the KB mirrors can be used with the different sample environments available on the beamline (e.g. magnets, spectroscopy chamber, ...).

At the time of writing, we are approaching the final design review. The mirror mount is designed to enable the beam size to be controlled remotely from a maximum size of 1 (H) x 1 (V) mm² down to a few microns.

The new capabilities that the KB mirror assembly adds ensures that users will be able to spatially map out the structural properties of a material sample while it is undergoing a functional process; charging/discharging of a battery material or operation of a catalyst material would be typical examples. Care has been made in the final design to ensure that we minimise the constraints to other operational modes that are employed on XMaS but also retain a degree of flexibility. The system is planned to be available for users from the April 2023 call.

[1] IDT (www.idtnet.co.uk)

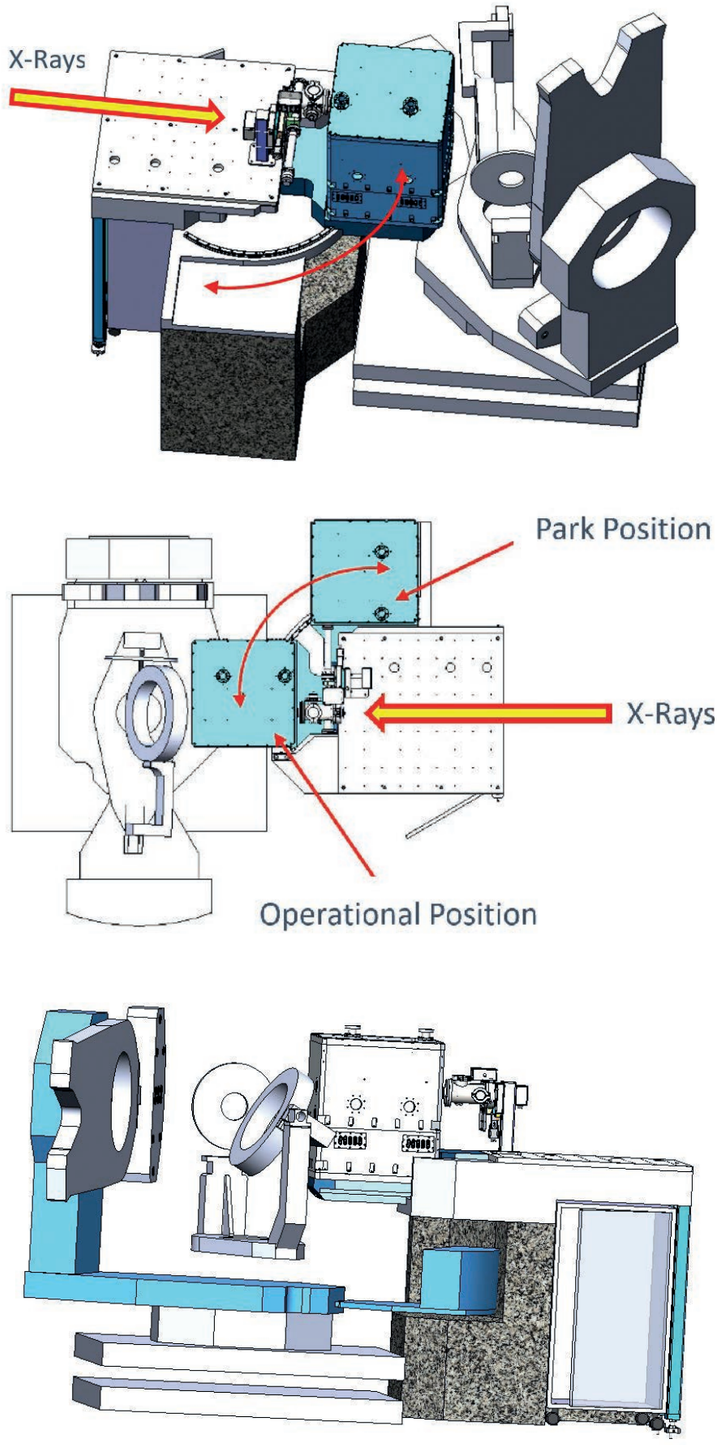

Fig. 7: The IDT designed KB mirror system envisioned for the beamline. To maximise flexibility, the system will be retractable and be positioned to the side of the main axis when the focused beam is in use.

Novel *in-situ* stress rig reveals origins of giant piezoelectric effect

M.G. Cain, P. Finkel, P. Thompson

Sonar technology was one of the first practical commercial applications of piezoelectricity. Today the active power of piezoelectric materials remains vital to the healthcare, defence, fishing, geophysical mapping and sea monitoring sectors. Due to extremely high piezoelectric coefficients, several times larger than those of commercial ceramics, piezoelectric single crystals are widely used in ultrasound transducers and now with the help of our measuring capabilities and materials science expertise, this technology is being pushed even further.

With an extraordinarily high piezoelectric coefficient and extraordinary coupling coefficient achieved by "domain engineering", i.e. cutting and poling the crystals along certain planes, novel $xPb(In^{1/2}Nb^{1/2}O_3$ - (1-x-y) $Pb(Mg^{1/3}Nb^{2/3})O_3$ - $yPbTiO_3$ (PIN-PMN-PT) "ternary" compositions explain how well these materials can convert mechanical (acoustic) energy in sonar transducers to electrical energy and how they could be used in this way in the future.

Particular to this PIN-PMN-PT class of single crystal, a recently discovered phenomenon of enhanced piezoelectricity can be linked to the presence of intermediate ferroelectric states in-between the stable phases normally seen using X-Ray Diffraction (XRD) that allows for enhanced polarisation rotation as the material is mechanically or electrically excited. It was an important feature, which needed clarification. The US Naval Research Laboratory worked with Electrosciences and the XMaS beamline to critically follow the changes in crystallography (Fig. 8) as the crystal was "squashed" (i.e. put under uniaxial stress) and under an applied electrical field (Fig. 9).

The Electrosciences ES1500 Multifunctional Stress Rig (jointly developed under ONRG and EU EMPIR funded programmes) was used to explore the transformation of the crystal structure with stress and applied electric field and the results provided clarity on the phases present [1]. Additionally, the data were correlated to the unusual optical properties of the material, which changed from translucent to transparent as it traversed the phase transition.

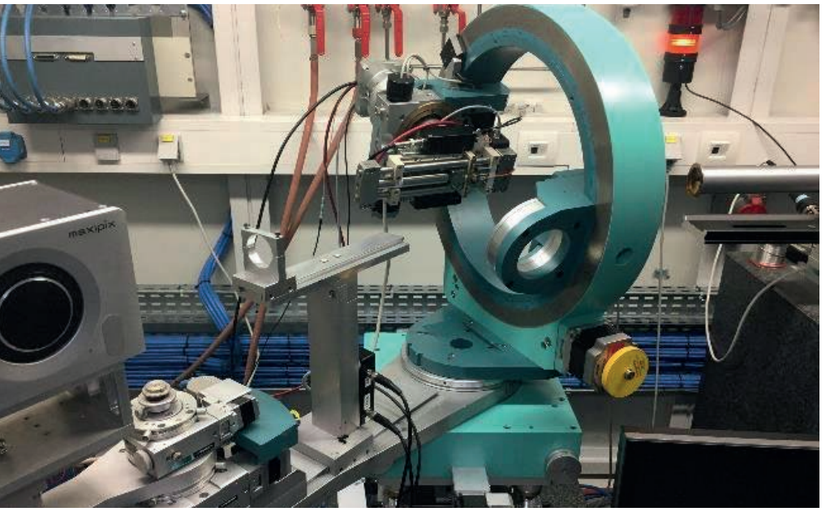

Fig. 8: XRD setup used in the offline X-ray source laboratory.

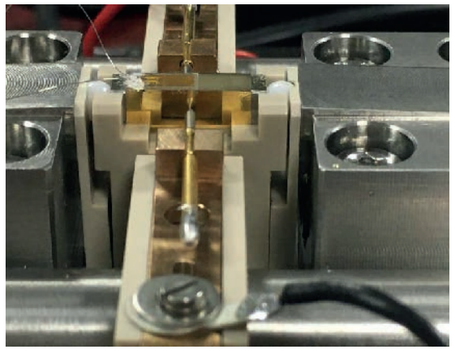

Fig. 9: PIN- PMN-PT sample mounted within the multifunctional stress rig. Mechanical force is applied horizontal while electric field is applied to the electroded sample via the two pins showed in the image.

Preliminary dynamic data were published in 2020 in "APL" [2], and the synchronised X-ray, optical, stress and field data have recently been published in November 2021 in "Advanced Materials" [1]. This collaborative work helped in the development of a new feature of the stress rig: incorporating an integrated optical transmission system to probe the structure, function and optical properties of materials simultaneously.

According to Peter Finkel, Lead Scientist at The Naval Research Laboratory, Washington DC, USA: *"The Electrosciences Stress Rig was instrumental in both papers and helped us refine our ideas of why these materials exhibit such high levels of piezoelectric activity. The measurements have also revealed a completely new feature of this class of material with applications that would benefit from stress-induced switching of optical signals!"*.

[1] P. Finkel *et al.*, Adv. Mat., 2106827, 1-8 (2022).
[2] P.A. Patterson *et al.*, Appl. Phys. Lett., 116, 222903 (2020).

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Quantum order by disorder revealed under magnetic field

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Ferromagnets are ubiquitous in everyday life, present in household items ranging from magnets on a fridge door to the sensors and motors inside it, with dozens present in an average car. As temperature is raised, ferromagnetic order disappears but, more rarely, the uniform magnetic order can first be replaced by a static magnetic wave. One mechanism to explain this is known as quantum order by disorder (QOBD); the wave (the order) forms because it has more low-energy excited states (the disorder) available to it than the uniform state and this lowers its energy, offsetting the energy cost of producing the wave.

The first observation of QOBD waves was reported when the ferromagnet PrPtAl was heated, just before it completely lost its magnetism [1]. Two modulated spin density wave states (SDW1 and SDW2) with different temperature dependent propagation vectors were observed to form over a small temperature interval. To investigate QOBD in more detail, Resonant Elastic X-ray Scattering (REXS) was carried out on a PrPtAl single crystal under applied magnetic fields at XMaS. REXS affords both magnetic site selectivity and high resolution, making it a powerful method for characterising modulated magnetism. We found that in a field, modulated order can be induced at temperatures extending down towards the absolute zero, accessing the quantum regime.

One of the most remarkable properties of the QOBD theory is that it explains order in which moments appear along magnetic hard axes [2]. In SDW1 and SDW2, this is manifest by the modulated moments having components along both the *a*-axis (easy-axis) and *b*-axis (hard-axis). For field applied along the easy axis, REXS showed that SDW1 and SDW2 are strongly suppressed by an extremely modest field of 0.02 T, in qualitative agreement with QOBD theory. Additionally, SDW1 was found to be strongly polarisable compared with SDW2, helping to explain why there are two states.

Applying a carefully aligned magnetic field along the hard b-axis had more striking consequences. SDW1 and SDW2 were found to survive up to 2 T, but then transformed to a newly discovered modulated state, SDW3 [3]. Ferromagnetism is thus completely enclosed by modulated states in the field-temperature phase diagram (Fig. 10), extending down to low temperature. The structure of SDW3, consistent with both experiment and theory, is a modulated fan with the moment direction oscillating around the hard *b*-axis. It lies at the boundary between ferromagnetism with moments along the a-axis and a field-polarised state with moments parallel to the field. Complementary magneto-transport and inelastic neutron scattering measurements provide compelling evidence that SDW3 is driven by QOBD theory [3].

In conclusion, PrPtAl is shown to provide an archetypal example for QOBD-induced modulated magnetism. For the first time, magnetic field has been used to tune a ferromagnet into a QOBD-modulated state at very low temperature. This may help us understand why other field-induced quantum phenomena, such as re-entrant superconductivity, can occur in other quantum materials, notably in ferromagnetic superconductors.

[1] G. Abdul-Jabbar *et al.*, Nat. Phys., 11, 321 (2015).
[2] M. Brando *et al.*, Rev. Mod. Phys., 88, 025006 (2016).
[3] C.D. O'Neill *et al.*, Phys. Rev. Lett., 126, 197203 (2021).

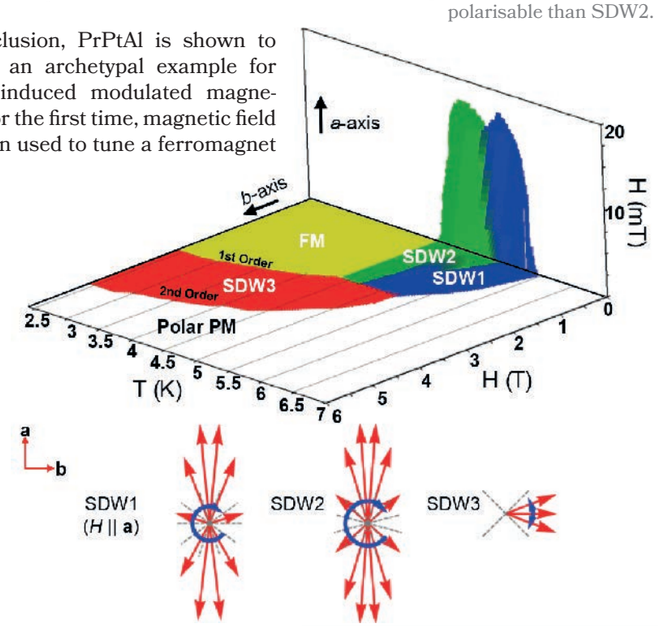

Fig. 10: Schematic temperature-field phase diagram for PrPtAl with fields applied along the easy *a*-axis (vertical) and hard *b*-axis (horizontal) based on REXS measurements. SDW1 (blue), SDW2 (green) and SDW3 (red) modulated states form a ridge in 3D around a ferromagnetic plane (yellow), across which the *a*-axis ferromagnetic moment reverses. Shown below this, is a projection of the moment directions viewed along the *c*-axis for one modulation period; SDW1 is shown in a small *a*-field to emphasise it is more strongly polarisable than SDW2.

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Substrate protection with sweet corrosion scale: Mind the gap

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Corrosion is an omnipresent threat in many engineering scenarios [1]. On this basis, significant effort continues to be applied to control this issue, including exploring more environmentally sustainable approaches. One such route is exploitation of the protection offered by naturally occurring corrosion scale, e.g. iron carbonate crystallites formed on iron/carbon steel in sweet (CO_2- saturated) aqueous solutions, which are encountered in geothermal power plants, carbon capture operations and oil production.

In the current work [2], we critically assess the corrosion protection afforded by iron carbonate scale, using a combination of electrochemical measurements and *operando* Grazing Incidence X-Ray Diffraction (GI-XRD). Fig. 11 shows a schematic of the custom-built cell employed in this study, which allows GI-XRD data to be acquired from a submerged sample: the ability to undertake measurements at low dissolved O_2 concentrations for a range of solution cell temperatures (~20°C to 80°C) are notable features of the design.

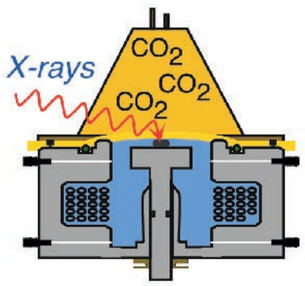

Fig. 11: Schematic of custom-built cell used in this work for combined electrochemical and *operando* GI-XRD measurements.

As illustrated by the sequence of diffractograms in Fig. 12, iron carbonate scale is found to grow on the surface of an iron sample in a sweet solution. Under the prevailing conditions (pH = 6.8, T = 80°C), a steady state is achieved after ~ 300 min of immersion, i.e. the iron carbonate signal no longer increases.

Based on previous work [3], the growth of the surface scale is expected to reduce the corrosion of the iron. This assumption was verified through determination of the corrosion rate from electrochemical measurements undertaken in parallel with GI-XRD data acquisition. It was concluded that the scale does significantly impede corrosion of the substrate, but that electrochemical iron dissolution (i.e. corrosion) from gaps between iron carbonate crystallites (Fig. 13) is higher than previously anticipated.

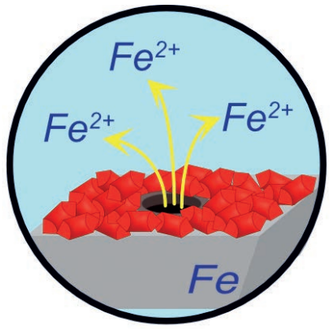

Fig. 13: Cartoon of localised electrochemical iron dissolution (Fe(s) → Fe^{2+}(aq)) from a gap between iron carbonate scale crystallites (shown in red).

This result has key implications for corrosion control using naturally occurring iron carbonate scale. In our opinion, even in its *intact* state (i.e. ignoring local breakdown), this scale is almost certainly, at best, semi-protective, and *in situ* chemical treatment and/or surface pre-treatment is required to prevent loss of structural integrity.

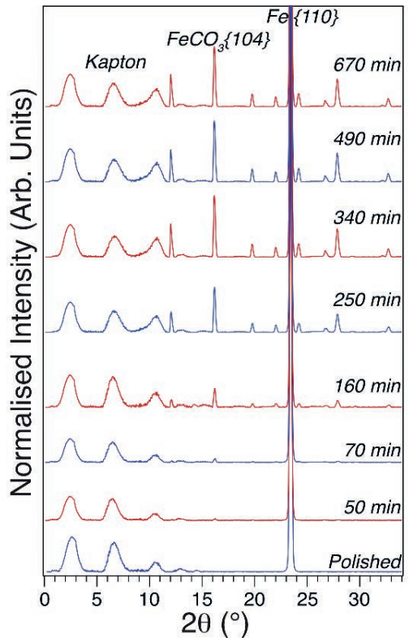

Fig. 12: Diffractograms, acquired as a function of immersion time, from an iron sample submerged in a sweet solution at pH = 6.80 ± 0.05, T = 80 ± 3°C and [O_2(aq)] < 20 ppb. The bottommost plot is of a polished substrate, acquired prior to immersion, which exhibits features attributable to iron and kapton; the latter signal arises from cell components located in front of the sample. Following immersion, each additional peak that appears can be assigned to iron carbonate ($FeCO_3$).

[1] G. Koch *et al.*, NACE 2016 Impact Study, International Measures of Prevention, and Economics of Corrosion Technologies Study (2016).

[2] M. Al Kindi *et al.*, ACS Appl. Mater. Interfaces, 13, 48, 58193 (2021).

[3] G.R. Joshi *et al.*, Corros. Sci., 142, 110 (2018).

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Wolfram's copper blisters

A.M. Korsunsky, L. Romano Brandt

How do diffusion processes and atomic intermixing happen at the nanoscale? Does the outcome differ from the macroscale in terms of mechanisms, rates and consequences? What methods can allow us to witness, follow, understand and predict these phenomena, for the benefit of formulating and developing new technologies?

In many binary metallic systems, the interaction between atoms creates chemical reactions in the solid state, thereby forming intermetallic compounds and phases with specific composition, structure, and properties. The copper (Cu) and tungsten (W) system represents an immiscible combination in which the attention can be focused on species migration. Cu/W nano-multilayers (NML) with different individual layer thickness and period were chosen. These may also provide insight into the operation and effectiveness of diffusion barriers in advanced electronics systems.

In the present study [1], Cu/W NML deposited on a Si substrate using ion beam were analysed using *in situ* GISAXS to determine the nanoscale evolution of their structure during heating. Post-treatment transmission EDX was used in our Oxford MBLEM lab to observe the consequences of the accelerated diffusion processes within buried layers. Further supporting techniques such as XRR, TEM, WAXS, and AFM were employed to develop a detailed picture of the multilayer structure before, during and after heating. It was found that the pronounced in-plane compressive residual stress and the defect population induced by ion beam deposition led to low thermal stability due to the thermally activated self-interstitial and vacancy diffusion. This process was tracked until the complete degradation of the layered structure at moderate temperatures. The formation of Cu protrusions was observed, and a model was formulated for stress-assisted Cu diffusion driven by Coble creep along W grain boundaries. Interaction with the Si substrate was also detected and incorporated into the analysis.

The model showed excellent agreement with the experimental data (Fig. 14) and provided the explanation for the observed strong correlation between thin film deposition conditions, microstructural properties, and low thermal stability. The experimental and modelling approaches developed and employed in this study can be readily and insightfully applied to other multilayer systems [1].

[1] R.L. Brandt *et al.*, ACS Appl. Mater. Interfaces, 13, 5, 6795 (2021).

AFM: Atomic Force Microscopy.

EDX: Energy Dispersive X-ray Spectroscopy.

GI-SAXS: Grazing Incidence Small Angle X-ray Scattering.

TEM: Transmission Electron Microscopy.

WAXS: Wide Angle X-ray Scattering.

XRR: X-ray Reflectometry.

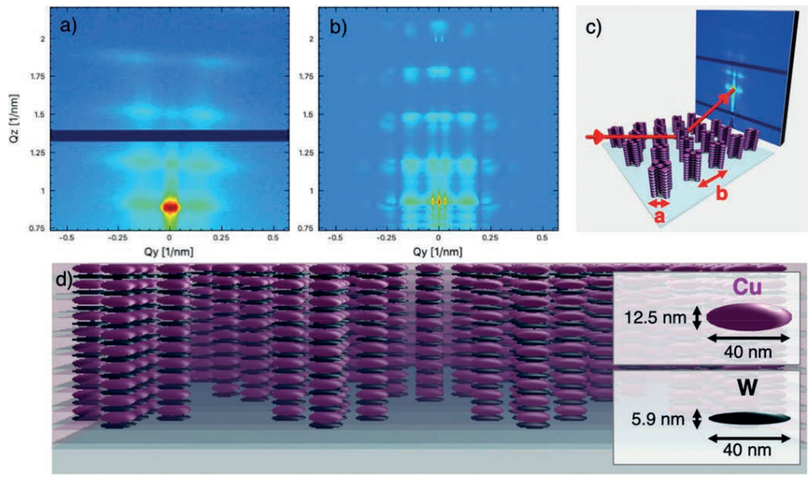

Fig. 14: (a) GI-SAXS pattern of Cu/W NML at 30°C. (b) Simulated Cu/W GI-SAXS pattern based on BornAgain material model. (c) Overview of BornAgain model and scattering geometry. Cu/W columnar grains are shown on the Si substrate, alongside the incoming and reflected beam. While the Cu and W particles are embedded in Cu and W layers in the simulation, they have been removed from the illustration for the purpose of visibility. (d) Side view of rendering showing the simulated multilayer microstructure with columnar Cu/W growth. Dimensions are indicated on the right.

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Dendrimer interactions with cell membrane models

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Dendrimers are branched polymeric nanoparticles with a range of biomedical applications due to the tunability of the size and functionality of their terminal branches. In particular, dendrimers are being developed as vehicles for drug and gene delivery since they have numerous internal cavities for the loading of hydrophobic drugs and can bypass easily the cellular membrane. However, the application of dendrimers has yet to be fully exploited, largely due to uncertainties in the exact mechanism for bypassing the cell membrane barrier and the subsequent cytotoxic effects that could be elicited by dendrimers.

Recent X-Ray Reflectivity (XRR) measurements conducted at the XMaS beamline **[1]** have shed light on dendrimer-membrane interactions. Here, lipid multilayers were combined with dendrimers at varying concentration and at different stages in the multilayer production. The effects of the dendrimers with terminal positive charges or hydrophobic groups on the structure of these stacked lipid membranes (multilayers) were studied for the first time.

The structure of the multilayers was evaluated using XRR (Fig. 15). The thickness of the membranes as well as parameters describing long-range disorder and fluctuations in membrane thickness, were analysed. It was found that adding dendrimer to the multilayers resulted in membrane thinning and more disordered multilayer structures. Larger hydrophobic dendrimers were found to cause greater disruption compared to smaller hydrophobic dendrimers. However, the smallest positively charged dendrimers caused the most pronounced membrane thinning. Increased concentration of dendrimers was also found to increase membrane thinning. The method of the dendrimer addition to the multilayers also affected the membrane structure. This indicated the possibility of more effective incorporation of dendrimers when added at the earliest stage of multilayer fabrication.

These results have demonstrated the complexity of dendrimer-membrane interactions and provide a fundamental understanding of how the properties of dendrimers affect their cell-entry and toxicity. This is crucial for the future biomedical applications of dendrimers as well as nanocomposite materials in which nanoparticles are added for enhanced properties and functionality.

[1] L.J. Fox *et al.*, BBA, 1865 (4), 129542 (2021).

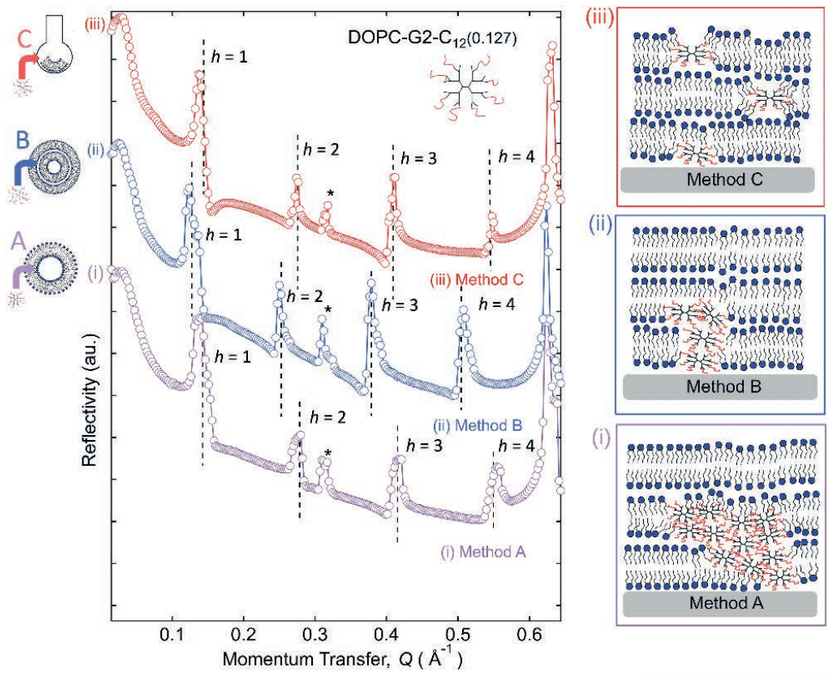

Fig. 15: XRR measurements of lipid multilayers prepared using three different methods (A,B & C), i.e. incorporating hydrophobic dendrimers at different stages. Analysis of the position (h=1,2,3,4) and width of the Bragg peaks enables evaluation of multilayer structural disorder.

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APPLICATIONS FOR SYNCHROTRON BEAM TIME

Two proposal review rounds are held each year. **Deadlines for applications to make use of the National Research Facility (CRG) time are normally 1st April and 1st October** for the scheduling periods August to February and March to July, respectively.

Applications for beamtime must be submitted electronically via the ESRF web page: www.esrf.eu. Select **"Users & Science"**, then choose **"Applying for beamtime"** from the drop-down list. On the right hand side, you can consult the instructions to submit your proposal and access the **"User Portal"**. Enter your surname and password and select **"Proposals/Experiments"**. Follow the instructions carefully — you must choose **"CRG Proposal"** and **"BM28 (XMaS - Mat.Sci.)"** at the appropriate stage in the process. If you experience any problems, please contact Laurence Bouchenoire (bouchenoire@esrf.fr). Technical specifications and instrumentation available are described on the XMaS web page (www.xmas.ac.uk). All sections of the form must be filled in. Particular attention should be given to the safety aspects with the name and characteristics of your samples completed carefully. Experiments requiring special safety precautions such as the use of electric fields, lasers, high pressure cells, dangerous substances, toxic substances and radioactive materials, must be stated clearly in the proposal. Moreover, any ancillary equipment supplied by the user must conform to the appropriate French regulations. Further information may be obtained from Martine Moroni, the ESRF Experimental Safety Officer for CRG beamlines (martine.moroni@esrf.fr, tel: +33 4 76 88 23 69). Please indicate your date preferences, including any dates that you would be unable to attend if invited for an experiment. This will help us to produce a schedule that is satisfactory for all.
When preparing your application, please consider that access to the National Research Facility is reserved for UK based researchers. Collaborations with EU and international colleagues are encouraged, but the proposal must be led by a UK based principal investigator. It must be made clear how any collaborative research supports the wider UK science base. Applications without a robust link to the UK will be rejected and should instead be submitted directly to the ESRF using their public access route.
Access to XMaS beamline is also available for one third of its operational time to the ESRF's user community. Applications for beamtime within that quota should be made in the **ESRF's proposal rounds (application deadlines 1st March and 12th September)**. Applications for the same experiment may be made to both XMaS directly and to the ESRF. Obviously, proposals successfully awarded beamtime by the ESRF will not then be given additional time in the XMaS allocation.
An experimental report on completed experiments must be submitted electronically, following the ESRF model. The procedure for submitting experimental reports follows that for the submission of proposals. Please follow the instructions on the ESRF's web pages carefully. **Reports must be submitted within 6 months of the experiment.** Note that the abstract of a publication can also serve as the experimental report! Please also remember to fill in the XMaS end of run survey form on completion of your experiment, which is available on the website (https://bit.ly/3JM6E7q).

Assessment of Applications
The independent Peer Review Panel considers the proposals, grades them according to scientific excellence, adjusts the requested beam time if required, and recommends proposals to be allocated beam time on the beamline. Experimental reports will also form part of the assessment criterion. Proposals which are allocated beamtime must meet ESRF safety and XMaS technical feasibility requirements. Following each meeting of the Peer Review Panel, proposers will be informed of the decisions taken and feedback provided.

APPLICATIONS FOR OFFLINE FACILITY TIME

Submit your application directly on the XMaS web site: www.xmas.ac.uk. Select **"XMaS Offline Facilities"** and then **"Application for Offline Facilities"**. Follow the instructions carefully and do not forget to upload your 1-2 page proposal at the end of the application form. Please contact the local staff to discuss any potential experiments. Successful offline proposals will be run as in-house experiments. We will complete the safety form with the information supplied in your application form as well as arrange site passes and any accommodation that may be required. As for synchrotron beamtime, offline users normally stay in the ESRF guest house or off-site hotels.

The XMaS facility implements transparent policies and procedures to guarantee that access is based on scientific excellence only. In partnership with the ESRF Safety office, we will endeavor to ensure that the facility can accommodate any user, but this may require an individual needs assessment. If you have any questions about accessing the facility at any stage of the application or experimental processes, please do not hesitate to get in touch.

Living allowances
These are €75 per day per beamline user — the equivalent actually reimbursed in sterling. XMaS will support up to 3 users per synchrotron experiment and only 1 on the offline laboratories. For experiments which are user intensive, additional support may be available. The ESRF hostel still appears adequate to accommodate all our users, though CRG users will always have a lower priority than the ESRF's own users. Do remember to complete the "A-form" when requested to by the ESRF, as this is used for hostel bookings, site passes and to inform the safety group of attendees.

Beamline people

ONSITE TEAM

Didier Wermeille
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is the Beamline Responsible who, in partnership with the Directors, oversees the activities of the user communities as well as the programmes and developments that are performed on the beamline. He is also the beamline Safety Representative. His expertise spans crystallography, high resolution diffraction, surface studies, magnetic scattering and electric field measurements.

Laurence Bouchenoire
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is the Beamline Coordinator. She looks after beamline operations and can provide you with information about the beamline, application procedures, scheduling, etc. Laurence should normally be your first point of contact. Her expertise is in magnetic scattering including polarisation dependence.

Oier Bikondoa
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is Beamline Scientist with expertise in soft matter materials, SAXS, GISAXS, GIWAXS, surface and reflectivity studies.

Edgar Giuterrez Fernandez
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is our new PDRA with expertise in SAXS.

Paul Thompson
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is the contact for instrument development, technical support, sample environments including electric field, liquid cells and catalysis. He is assisted by **John Kervin** jkervin@liv.ac.uk, who is based at the University of Liverpool but provides further technical back-up and spends part of his time on-site at XMaS.

PROJECT DIRECTORS

Chris Lucas clucas@liv.ac.uk and **Tom Hase** t.p.a.hase@warwick.ac.uk continue to travel between the UK and France to oversee the operation of the beamline.

Malcolm Cooper
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remains involved in the beamline operation as an Emeritus Professor at the University of Warwick.

Yvonne Gründer
yvonne.grunder@liverpool.ac.uk
joined the management team at Liverpool to provide additional support. She also oversees impact activities.

Sarah Jarratt
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and **Julie Clark**
Julie.Clark@liverpool.ac.uk
are the administrators on the project, based in the Department of Physics at Warwick and Liverpool, respectively. Sarah is the point of contact for user T&S claims and co-ordinates the annual XMaS Scientist Experience.

THE PROJECT MANAGEMENT COMMITTEE

The current membership of the committee is as follows:

P. Hatton (chair), University of Durham,
S. Crook, EPSRC,
M. Alfredsson, University of Kent,
M. Cain, Electrosciences Ltd,
A. Beale, University College London,
K. Edler, University of Bath,
B. Hickey, University of Leeds,
S. Langridge, ISIS, Rutherford Appleton Laboratoy,
C. Nicklin, Diamond Light Source,
W. Stirling, Institut Laue Langevin.

In addition to the above, the directors, the chair of the Peer Review Panel, the CRG Liaison M. Hahn and the beamline team are in attendance at the meetings which happen twice a year.

THE PEER REVIEW PANEL

The current membership of the panel is as follows:

R. Johnson (chair), University College London,
A. Hector, University of Southampton,
E. Heeley, Open University,
M. Skoda, ISIS,
R. Walton, University of Warwick.

In addition, either Chris Lucas or Tom Hase attends their meetings in an advisory role.

PUBLISH PLEASE!!... and keep us informed

One of the important XMaS KPIs is the number and quality of publications. We ask you to provide Sarah Jarratt (sarah.jarratt@warwick.ac.uk) with the reference and DOI whenever a new paper is published. Alternatively, you can submit your new publication reference directly through a form on our web site (https://bit.ly/2Gja4zX). Please also let us know about other impact generated as a result of XMaS work.

IMPORTANT!
It is important that we acknowledge the support from EPSRC in any publications. When beamline staff have made a significant contribution to your scientific investigation you may naturally want to include them as authors. Otherwise we ask that you add an acknowledgement of the form:

"XMaS is a UK national research facility supported by EPSRC. We are grateful to all the beamline staff for their support."

Editor: Laurence Bouchenoire - Graphic Designer: Sylvie Quercia - Print: WarwickPrint - March 2022

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